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Test Methods for Determining the Chemical Waste Compatibility of Synthetic Liners

Little (Arthur D.), Inc., Cambridge, MA

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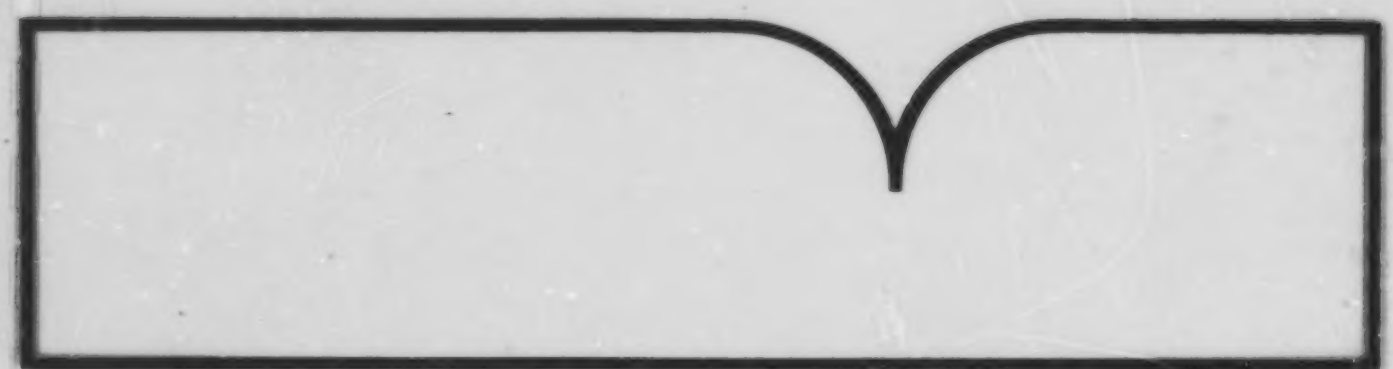

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TEST METHODS FOR DETERMINING THE CHEMICAL WASTE COMPATIBILITY OF SYNTHETIC LINERS

by

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16. ABSTRACT

A search was made for test methods for the chemical resistance of membrane liners (geomembranes). Disclosed methods and procedures were examined and compared. Two tests being promoted for general acceptance are NSF Standard No. 54 (a voluntary industry-generated test) and the proposed EPA Test Method 9090. Several other tests developed by liner manufacturers and researchers were found, as well as those methods generally applied to pipes, bottles, film, plastics, rubber sheeting and the like. Although details of tests vary, all are laboratory tests in which selected physical properties of the membrane are compared and evaluated after contact with the liquid for specified periods of time. All are tedious, time-consuming, and potentially costly. Useful data for product specification and application are derived from these tests, but none adequately addresses all issues and questions raised, especially with regard to liner life-time prediction. Nor is any one test universally accepted for use. A combination of compatibility tests (e.g., immersion, stress-crack, and permeation) may be necessary to fully characterize chemical resistance in specific cases. A superior test(s) based on a comprehensive understanding of liner compatibility remains to be developed.

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ABSTRACT

Flexible membrane liners (geomembranes) used to contain liquid chemicals and leachate at waste containment sites are required to be chemically resistant (compatible) to the liquid. In order to select a liner for use as well as judge its long-term reliability, its chemical resistance against the liquid(s) to be contained must be known. The measurement of compatibility is a complex matter, because a variety of physical and chemical interactions can occur, and compatibility failure of a membrane has never been adequately defined for this application.

A search was made for test methods that would ascertain the compatibility performance of liners. Disclosed methods and procedures were examined and compared. Two tests being promoted for general acceptance are NSF Standard No. 54 (a voluntary industry-generated test) and the proposed EPA Test Method 9090. Several other tests developed by liner manufacturers and researchers were found, as well as those methods generally applied to pipes, bottles, film, plastics, rubber sheeting and the like.

Although details of tests vary, all are laboratory tests in which selected physical properties of the membrane are compared and evaluated after contact with the liquid for specified periods of time. All are tedious, time-consuming, and potentially costly. Useful data for product specification and application are derived from these tests, but none adequately addresses all issues and questions raised, especially with regard to liner life-time prediction. Nor is any one test universally accepted for use. A combination of compatibility tests (e.g., immersion, stress-crack, and permeation) may be necessary to fully characterize chemical resistance in specific cases. A superior test(s) based on a comprehensive understanding of liner compatibility remains to be developed.

Proposed are two levels of effort (immediately practical, and long-term) directed at evaluating membrane compatibility. In the first, a test methodology based on current protocols and methods would be developed to provide three kinds of required information: short-term (testing up to 30 days' exposure), intermediate (testing up to 4 months' exposure), and long-term (greater than 4 months' exposure). In the second, research and test method development would be pursued with the purpose of exploring new methods, techniques, apparatus, etc., for better compatibility characterization.

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This report was prepared by Arthur D. Little, Inc., Cambridge, Massachusetts, under a contract with the Monitoring and Data Support Division of the Office of Water Regulations and Standards, Environmental Protection Agency. It was designed to contribute to research studies and evaluation of the use of flexible membrane liners (FML) in hazardous waste disposal sites. We gratefully acknowledge the guidance given to us by our Work Order Manager, Robert Hartley, and several other individuals, especially Robert Landreth of the Office of Research and Development, Municipal Environmental Research Laboratory. These individuals, plus Henry Haxo, Jr. (Matrecon, Inc.) and FML industry representatives, provided valuable information and comments that contributed to the success of this effort.

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CONTENTS

DISCLAIMER AND PEER REVIEW NOTICE		ii
ABSTRACT		iii
ACKNOWLEDGMENTS		iv
CONTENTS		v
FIGURES		vii
TABLES		viii
1.0	INTRODUCTION	1
	1.1 BACKGROUND	1
	1.2 NEED FOR REVIEW AND ASSESSMENT	2
	1.3 PURPOSE AND SCOPE	2
2.0	SUMMARY OF FINDINGS	4
	2.1 CURRENT TEST METHODOLOGY AND LIMITATIONS	4
	2.2 CONCLUSIONS	4
3.0	RECOMMENDATIONS	7
4.0	GENERAL TECHNICAL CONSIDERATIONS	9
	4.1 NATURE OF LINER MATERIALS	9
	4.2 NATURE OF CHEMICAL/LEACHATE	10
	4.3 NATURE OF CHEMICAL COMPATIBILITY	16
	4.4 COMPATIBILITY MEASUREMENT FEATURES	17
5.0	PRESENT COMPATIBILITY TESTS	19
	5.1 BACKGROUND	19
	5.2 TEST METHODS	20
	5.3 COMPARISON OF TEST METHODS	22
	5.3.1 ISO 175 - Plastics - Determination of the Effects of Liquid Chemical including Water	28
	5.3.2 ISO 1817 - Vulcanized Rubbers - Resistance to Liquids	30
	5.3.3 DIN 53 521 - Determination of Resistance to Liquids, Vapors, and Gases	31
	5.3.4 DIN 53 532 - Permeability of Elastomer Sheeting to Liquid Fuels	33
	5.3.5 BS 4618 - Chemical Resistance of Plastics to Liquids	34
	5.3.6 BS 5173 - Hoses - Chemical Resistance Tests	36
	5.3.7 EPA Method 9090 - Compatibility Test for Wastes and Membrane Liners	37
	5.3.8 NSF Standard 54 Flexible Membrane Liners	40
	5.3.9 ASTM D543 (78) - Resistance of Plastics to Chemical Reagents	43
	5.3.10 ASTM D814 - Rubber Property - Vapor Transmission of Volatile Liquids	45
	5.3.11 ASTM G20 - Chemical Resistance of Pipeline Coatings	46
	5.3.12 ASTM D471 - Rubber Property - Effect of Liquid	48

CONTENTS (continued)

5.3.13	MIL-T-6396D Aircraft Tanks - Fuel, Oil, Water-alcohol, Coolant	50
5.3.14	Schlegel Test for HDPE	51
5.3.15	J.P. Stevens Test	53
5.3.16	Gundle Test	54
5.3.17	Simulation Test (Haxo)	55
5.3.18	Pouch Test (Haxo)	56
5.3.19	Tub Test (Haxo)	57
5.3.20	Immersion Test (Haxo)	58
5.3.21	NSF's FML Project	59
5.3.22	Harwell Assessment of Two HDPE Landfill Liners by Application of an Accelerated Test	61
5.3.23	Sequential Chemical Absorption Techniques for Evaluating Elastomers	63
5.3.24	Guide to Fluid Resistance of Rubber and Elastomers	64
5.3.25	Environmental Stress-cracking by Creep Rupture Tests	65
5.3.26	Chemical Stress Relaxation Test	66
5.3.27	A New Method for Determining Environmental Stress-crack Resistance of Ethylene-based Plastics	68
5.4	TEST STATE-OF-THE-ART	70
5.5	CONSENSUS MEETING	71
5.6	MINUTES OF MEETING ON COMPATIBILITY TEST METHODS AND FML REQUIREMENTS	72
5.6.1	Exposure Conditions	72
5.6.2	Test Methods	74
5.6.3	Cost Profile	75
5.7	TEST EQUIPMENT AND COST CONSIDERATIONS	76
6.0	GENERAL APPROACH TO COMPATIBILITY TESTING	78
6.1	THE SCOPE OF TESTING	78
6.1.1	FML Material versus Liquid Challenge	78
6.1.2	Parameters	80
6.1.3	Measurements and Observations	81
6.1.4	Test Details	82
6.2	THE CURRENT DILEMMA AND ROUTES TO RESOLUTION	83
REFERENCES		91
APPENDIX A		97

FIGURES

Number		Page
5.1	ISO 175	29
5.2	DIN 53 521	32
5.3	BS 4618	35
5.4	METHOD 9090	39
5.5	NSF STANDARD 54	42
5.6	ASTM D543	44
5.7	ASTM G20	47
5.8	ASTM D471	49
5.9	SCHLEGEL	52
5.10	NSF's FML PROJECT	60
5.11	HARWELL REPORT	62
5.12	CHEMICAL-STRESS RELAXATION	67
5.13	ENVIRONMENTAL STRESS-CRACKING OF ETHYLENE PLASTICS	69
6.1	THE COMPATIBILITY TEST SCHEME	79
6.2	DETAILED SECTION THROUGH A VESSEL	85
6.3	STRESS RELAXATION CLASSIFICATION	86
6.4	DMA - UNEXPOSED HDPE	88
6.5	DMA - EXPOSED HDPE	89

TABLES

Number		Page
4.1	LABORATORY TEST CHEMICALS	11
4.2	MISCELLANEOUS TEST PRODUCTS	12
4.3	COMPOSITION OF THREE SELECTED LANDFILL LEACHATES	13
4.4	POTENTIAL ORGANIC CHEMICALS IN WASTE LIQUIDS	14
4.5	AVERAGE DATA FROM SEVEN DIFFERENT INDUSTRIAL LEACHATES	15
5.1	KEY WORDS USED IN LITERATURE SEARCH	21
5.2	INTERNATIONAL STANDARDS AND TESTS	23
5.3	NATIONAL STANDARDS AND TESTS	24
5.4	INDUSTRIAL STANDARDS AND TESTS	25
5.5	PROJECT TESTS	26
5.6	SELECTED ACADEMIC/LITERATURE TESTS	27
5.7	TENSILE AND TEAR TESTING PROTOCOLS	38
5.8	TEST METHODS	41

1.0 INTRODUCTION

1.1 BACKGROUND

The U.S. Environmental Protection Agency (EPA) published interim final regulations for the land disposal of hazardous wastes on July 26, 1982. These regulations became effective on January 26, 1983. The regulations specify design and operating standards relating to the use of liner and cap systems for the purpose of waste containment.

While the liner and cap systems may be based on compacted clays, admixtures (e.g., concrete and asphalt), and soil sealants, liners fabricated from synthetic polymer materials (often referred to as flexible membrane liners - FML*) will be used at a large number of the waste sites. The preamble to 40 CFR, Parts 122, 260, 264, and 265 (U.S. EPA, 1982), states that "in the cases of landfills and of surface impoundments used to dispose of hazardous waste, the regulations provide that the liner must be constructed of materials that prevent wastes from passing into the liner. Synthetic liners are the only commonly used materials of which EPA is aware that would meet this standard."

Since the primary function of a liner is to contain waste liquids, a priori, a liner must be resistant to the liquid. A key issue in the use of an FML is its compatibility (chemical resistance)* with the waste liquid it will hold. The criteria for evaluating chemical resistance and the choice of test methods are part of that issue.

Although compatibility data derived from field tests constitute actual use data, most data, by necessity, are obtained from laboratory tests. Field information is preferred because it reflects "real" exposure. As a matter of fact, adequate field data have not been generated because the use of FML for waste containment is too new a technology (in the order of 20 years of experience). Although field information is currently being generated, field testing is a slow process. To expedite the evaluation and selection of an FML, data are obtained from laboratory tests in which an FML is exposed to a chemical challenge under set conditions, and then properties--usually physical--of the exposed FML are measured. Laboratory tests serve as the primary screening approach to FML chemical compatibility evaluation.

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*The currently used term "geomembrane" includes FML. In FML technology, a "compatible" liner is generally accepted as one that is "resistant" to chemical attack as judged by its changes in physical properties upon exposure to liquid waste.

A detailed introduction to the FML industry, liner issues, and testing in general, including chemical compatibility, is found in other sources (Refs. 1-4).

1.2 NEED FOR REVIEW AND ASSESSMENT

For the benefit of regulators, planners, and designers, the current test methodology requires review and assessment so that uncertainties about data can be minimized. Chemical resistance data are needed for FML product development, screening purposes, site liner-waste matching, and liner life prediction. Although standard compatibility tests are being proposed or promulgated (e.g., NSF 54 and EPA 9090), a variety of other tests available within the FML industry and/or plastics and rubber industries are currently in use. The meaning and value of data derived from the tests are unclear because of differences in technique. All the applicable tests have never been compared to see how they differ, or if one is more informative than another. This report attempts to review identified compatibility tests pertinent to FMLs and assesses them in relation to chemical resistance measurement and the required needs for FML products.

1.3 PURPOSE AND SCOPE

The purpose of this assignment was to compile, review, and evaluate available test methods for measuring and/or estimating the chemical compatibility of polymeric flexible membrane liners (FML) with liquid wastes for use at hazardous waste treatment, storage, and disposal facilities.

The intention was not to write a test method for FML/waste compatibility, but to assess the current state of affairs and direct the thinking of all those concerned toward routes that might lead to a satisfactory, comprehensive, and reliable chemical compatibility measurement technique.

In carrying out this task, the focus has been placed on identifying and describing not only tests used to assess FMLs, but also tests from related technologies that are used to evaluate chemical compatibility. The investigation included a broad range of tests, including industry, academic, and research tests, and looked at both presently used and proposed methods. Arthur D. Little's extensive in-house data bank was examined, a literature search was conducted, and many individuals and organizations were contacted in both the United States and abroad. Included among these contacts were Government agencies (Federal and international), investigators and researchers in the field, standards and testing organizations, and FML manufacturers and suppliers.

Finally, a meeting was held at Arthur D. Little, Inc., in Cambridge, Massachusetts. In attendance were industrial and Government experts in FML technology and testing. A goal of this meeting was to develop a consensus about compatibility testing.

2.0 SUMMARY OF FINDINGS

2.1 CURRENT TEST METHODOLOGY AND LIMITATIONS

Current test methodology has evolved from metal corrosion testing and rubber and plastic testing. All current tests, even with their variation in details, fall into a pattern of contacting the membrane with the liquid for specified periods of time, followed by examination of the FML appearance, measurement of weight and dimensional change, as well as measurement of stress-strain properties (e.g., tensile strength, elongation, modulus). Alternative methods of exposure include simple suspension of the FML in liquid, one-side liquid contact of the membrane fixed in a cell, exposure in a dish to simulate ponding, and pouch testing in which waste is enclosed in the FML. Hardness and puncture resistance may also be measured. Stress-cracking, an important failure mode for some polyethylene-based membranes, may be measured as well. Because no good test method exists for tear resistance, this property is difficult to deal with, especially in the case of fabric-reinforced FML. However, tear resistance is often measured.

There are problems with current methodology. Most tests produce only indirect indication of FML chemical resistance, and important parameters are neglected or minimized in FML product evaluation. No actual determination of chemical change is made in routine testing. In the absence of true long-term data from field or laboratory (the FML technology is too new), current compatibility data, in conjunction with industrial experience, must be used to judge future behavior of FML. Lack of correlation with field performance remains a present issue. The experience baseline with complex waste compositions and their often unpredictable behavior is limited, and current physical tests may measure properties that are only partial indicators of actions taking place between membrane and liquid. Although attention in testing is focused on the chemical constituents of waste, the role of water (an active aggressor and catalyst by itself) is frequently overlooked or underestimated. Mass transfer of liquid (permeation) through an FML is not often measured and, until recently, has not been considered an important parameter for FML product evaluation. Permeation testing is considered separately from compatibility testing; it involves different test procedures.

2.2 CONCLUSIONS

1. Presently there is no generally accepted test method that fully meets the needs of industry or regulatory agency for the chemical compatibility assessment of FML in the presence of waste liquid.

2. Only two general test methods, recently introduced, deal specifically with chemical compatibility testing of membrane liners. They are: NSF Standard 54 and EPA Method 9090.* NSF 54 offers a recommended test method for long-term performance of membrane liners in a chemical environment. The "long-term" evaluation is based on extrapolation from very short-term measurement. Method 9090, a proposed method, is used in determining the effects of chemicals in a pit, pond, lagoon, or landfill-type installation on the physical properties of FML materials intended to contain them. Both tests evaluate chemical compatibility indirectly by the measurement of changes in several physical properties after exposure. Both tests incorporate standard ASTM methods for testing mechanical properties of rubber and plastics. Neither test deals directly with permeability nor environmental stress-cracking. (The NSF document contains a stress-crack requirement in the specifications, but it is not part of compatibility evaluation.) Because of sampling requirements, the more elaborate test protocol, the need for special exposure apparatus, and longer specified time for testing, Method 9090 is a more complex test than NSF 54 and would be more costly to run.

3. Chemical compatibility testing in the laboratory is conducted with actual leachate, waste, or reagent. Reagent testing is preferred for screening, but all current methods specify exposing the test specimen to actual waste samples or leachate. Exposing test specimens in an actual waste containment facility is recommended, and provision for inclusion of membrane coupons at the site for periodic examination for compatibility appears to be an obvious prerequisite for obtaining real data over the long term. Chemical class lists versus the FML type developed by industry and investigators for tests appear to be adequate for the initial stages of selecting an FML for a specific site.

4. FML chemical compatibility testing is made complex because of a large variety of possible interactions among components of the process. Figure 6.1 (p. 79) gives a schematic overview of this complexity.

5. Compatibility testing is tedious, time-consuming, costly, and potentially dangerous due to the need for handling toxic substances. The ideal route to testing has not yet been established, and long-term prediction is tenuous because of incomplete knowledge.

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*A revised version of Method 9090 was released by EPA in October, 1984. See NOTE on p 37.

6. Current tests, including those discussed above, deal with only part of the compatibility problem. The broader issue includes questions about practicability, cost, time span, significance, and reliability.

7. It appears that there will be no immediate and ideal resolution of the FMI chemical compatibility test issue. It is obvious that NSF 54 and Method 9090 are the current contenders for a standard test method, since other tests have not been developed sufficiently for FML evaluation. However, whether NSF 54 is followed, Method 9090 is imposed, other methods surface, or industry goes its own way, the problems are too diffuse and complex to rely on a simple set of rules as presently conceived. The crux of the problem is related to the need for good long-term data in what is essentially a new industry, and the fact that chemical compatibility indicators remain undefined. Truly reliable compatibility data will be generated with time, but continuous action is required by industry, regulatory agencies, and the research community to define the requisite parameters for evaluation, and then to develop the appropriate tests.

3.0 RECOMMENDATIONS

Effort on two levels is needed to evaluate FMLs for waste liquid containment. The first level of effort deals with the immediate practical issue of ascertaining FML compatibility for a particular application. The second--of long-term nature--focuses on understanding FML compatibility in the chemical/physical context, and then devising test methods for measuring the identified parameters. The first relies on maximizing application of current tests. The second explores new or untested methods.

For the first approach, it is recommended that test protocol be developed that would provide three kinds of information which might satisfy industry and regulatory body needs to the different degrees required:

I. Short-term tests (up to 30 days' exposure),
II. Intermediate tests (up to 4 months' exposure),
III. Long-term tests (greater than 4 months' exposure).

Level I data would be used to screen FML/liquid combinations for FML development and for selection purposes; Level II data would provide significantly more data related to expected life and real performance; and Level III data would provide confirmation of compatibility and correlation with performance goals.

All three levels of testing can be conducted with known current or proposed methodology. Details of testing might vary, but sufficient guidelines are already in place in the currently cited tests. Membrane actually exposed in the waste site should be tested to confirm laboratory evaluation. In addition to the basic physical testing, stress-crack measurements should constitute an additional requirement for testing polyethylene-based polymer membranes. The test matrix in Section 5.6.2 (p. 74) summarizes requirements for physical testing of current FML.

The approach to testing using current methods discussed in the text (e.g., EPA 9090, NSF 54, ASTM D543, ASTM D471, ISO 175, DIN 53-521, BS 4618, etc.) should be recognized as providing only an abbreviated assessment of chemical compatibility. Other properties not now part of a standard compatibility test may be useful or even better indicators. In the second approach to establishing the chemical resistance of FML, an exploratory course is called for in which FML properties are evaluated with techniques not now employed for compatibility measurement. Information about visco-elastic behavior and permeability obviously should be included. Methods for measurement of dynamic stress-strain properties, dynamic mechanical analysis, torsional stress, and thermal properties, especially under stress or load, are examples of techniques that might be explored further.

It is recommended that, besides pursuing practical compatibility testing on the three levels above, research be conducted to evaluate other techniques and approaches.

4.0 GENERAL TECHNICAL CONSIDERATIONS

Before dealing with specific test methods, a variety of general technical considerations should be reviewed. It is important to be aware of these factors that play a role in determining chemical compatibility, so that test methods might be better assessed later.

4.1 NATURE OF LINER MATERIALS

Commonly available industrial liner products are constituted principally of the following chemical polymeric materials:

Polyvinyl chloride (PVC);
Polyethylene, low-density (LDPE) and high-density (HDPE);
Chlorinated polyethylene (CPE);
Chlorosulfonated polyethylene (CSPE, CSM);
Ethylene propylene rubber (EPDM);
Epichlorohydrin rubber (ECO);
Neoprene rubber (CR);
Butyl rubber (IIR)
Asphalt/bitumen (A/B).

Asphalt/bitumen and admixes with inorganic aggregate and other polymers, although useful in water containment, play a minor role in hazardous waste containment. They are not considered in this document due to lack of information about them relative to test methods.

All the above polymeric materials and FML products made from them vary in degree of resistance to a specific chemical. A compendium of currently available compatibility data is being prepared in another study (Ref. 5).

It is important to understand that these polymers are compounded alone and in blends with additional materials and chemicals to achieve specific liner properties such as flexibility, ozone and UV resistance, and oil or water resistance. The liner membrane may vary in thickness (e.g., 30-120 mils), may be pigmented in different colors, may be a laminate, or may be reinforced with fiber scrim. Reinforcing fiber is usually polyester or nylon. Nevertheless, in trade parlance, the chemical polymer (the major constituent) is used to identify the type of liner. From a practical point of view, within each class of polymer manufacturers have made available a range of grades that are interactively overlapping with other polymers.

Although the behavior of a compounded FML will depend upon its total composition, the fundamental behavioral element will be the polymer phase. Therefore, the polymer has to be selected for its chemical resistance in a specific application. Chemical resistance will depend not only upon the chemical makeup of the polymer, but also

on other factors, such as degree of polymerization or molecular weight, degree of crosslinking, crystallinity, morphology, and the like.

Polymer chemistry and properties are discussed in many textbooks on the subject and need not be detailed here. In addition, product literature is available from manufacturers and suppliers. For a concise, state-of-the-art treatment of polymer chemistry, the reader is referred to the literature (Ref. 6).

4.2 NATURE OF CHEMICAL/LEACHATE

The nature and composition of the waste liquid challenging the liner may be infinite in variation. While water and selected organic liquids can be chosen to challenge the membrane alone, combinations of liquids and other waste material may also be used. Dissolved salts and other materials may be found in the challenge liquid. In the field, actual leachate/waste at a site is usually a complex mixture of many components. It may be difficult to analyze and characterize because of multiphase components or immiscible material. In any case, mixtures frequently do not behave in a manner predicted from components.

A number of the test procedures specify the liquids to be used. Not all tests use the same liquids. To illustrate, Tables 4.1 and 4.2 describe liquids specified in ISO 175 (International Standard-Plastics--Determination of the Effects of Liquid Chemicals, including Water).

Leachate represents an actual liquid composition apt to be experienced in the field. However, leachate components may change over a period of time, and the composition of the leachate does not remain constant. No standard leachate for testing has been identified or agreed upon. Industrial wastes may be acidic or alkaline, oxidizing or reducing, or may be brine, or oily, and contain heavy metals, pesticides, and such. Waste liquids may be classified as aqueous-inorganic, aqueous-organic, organic, or sludge.

Leachate composition and characteristics have been investigated and are reported elsewhere. Shuckrow *et al.* review information on leachate (Ref. 7) and TRW has compiled comprehensive data on hazardous waste leachate (Ref. 8). The TRW study covers 30 different leachates from some 11 landfills. Leachate is discussed in conjunction with membranes by the EPA (Ref. 9). Leachate *per se* is considered to be a dilute solution of inorganic and organic components in water. It may be acidic or basic, act as an oxidizing or reducing agent, or function as a solvent or plasticizer. Tables 4.3, 4.4, and 4.5 illustrate the complexity of leachate and the type of components that might be expected in the liquid. Other combinations of ingredients can be found.

TABLE 4.1. LABORATORY TEST CHEMICALS

Test Liquid	Concentration %(wt.)	Concentration kg/m³	Comments
Acetic acid	99.5	--	Concentrated
Acetic acid	5	50	Add 50 ml of concentrated acetic acid to 950 ml water
Acetone	100	--	--
Ammonium hydroxide solution	25	230	Expressed as ammonia (NH_3)
Ammonium hydroxide solution	10	96	Expressed as ammonia (NH_3)
Aniline	100	--	--
Chromic acid solution	40 (as CrO_3)	550	Add 3 ml of concentrated sulphuric acid per liter of solution
Citric acid solution	10	100	--
Diethyl ether	100	--	--
Distilled water	100	--	--
Ethanol	--	770	96% (V/V) (71° O.P.)
Ethanol	50	460	1000 ml of 96% (V/V) ethanol and 740 ml water
Ethyl acetate	100	--	--
n-Heptane	100	--	--
Hydrochloric acid	36	--	Concentrated
Hydrochloric acid	10	105	Add 250 ml of concentrated hydrochloric acid to 750 ml of water
Hydrofluoric acid	40	450	--
Hydrogen peroxide	30	330	Not diluted
Hydrogen peroxide	3	31	10 volumes of 30% (V/V) H_2O_2 and 90 volumes of water
Lactic acid	10	100	--
Methanol	100	--	--
Nitric acid	70	--	Concentrated
Nitric acid	40	500	Add 500 ml of concentrated nitric acid to 540 ml of water
Nitric acid	10	105	Add 105 ml of concentrated nitric acid to 900 ml of water
Oleic acid	100	--	--
Phenol solution	5	50	--
Sodium carbonate solution	20	216	Expressed as $Na_2CO_3 \cdot 10H_2O$
Sodium carbonate solution	2	20	--
Sodium chloride solution	10	108	--
Sodium hydroxide solution	40	575	--
Sodium hydroxide solution	1	10	--
Sodium hypochlorite solution	10	--	9.5% active chlorine
Sulphuric acid	98	--	Concentrated
Sulphuric acid	75	1250	Add 695 ml of concentrated sulphuric acid to 420 ml of water
Sulphuric acid	30	366	Add 200 ml of concentrated sulphuric acid to 850 ml of water
Sulphuric acid	3	--	Add 17 ml of concentrated sulphuric acid to 990 ml of water
Toluene	100	--	--
2,2,4-Trimethylpentane (iso-octane)	100	--	--

Source: ISO 175 (Ref. 25).

TABLE 4.2. MISCELLANEOUS TEST PRODUCTS

Test Liquid	
Mineral oil	Soap solution
Insulating oil	Detergent
Olive oil	Essence of turpentine
Cotton seed oil	Kerosene
Solvent mixtures	Petrol (gasoline)

Source: ISO 175 (Ref. 25).

TABLE 4.3. COMPOSITION OF THREE SELECTED LANDFILL LEACHATES

Constituent	Concentration (mg/l)		
Total solids	36,250	12,500	--
Volatile suspended solids	--	76	--
Total suspended solids	--	85	--
Total volatile acids as acetic acid	--	9,300	333
Acetic acid	--	5,160	--
Propionic acid	--	2,840	--
Butyric acid	--	1,830	--
Valeric acid	--	1,000	--
Organic nitrogen as N	--	107	--
Ammonia nitrogen as N	950	117	862
Kjeldahl nitrogen as N	1,240	--	--
pH	6.2	5.1	6.9
Total alkalinity as $CaCO_3$	8,965	2,480	--
Total acidity as $CaCO_3$	5,060	3,460	--
Total hardness as $CaCO_3$	6,700	5,555	--
Chemical and metals:			
Arsenic	--	--	0.11
Boron	--	--	29.9
Cadmium	--	--	1.95
Calcium	2,300	1,250	354.1
Chloride	2,260	180	1.95
Chromium	--	--	<0.1
Copper	--	--	<0.1
Iron	1,185	185	4.2
Lead	--	--	4.46
Magnesium	410	260	233
Manganese	58	18	0.04
Mercury	--	--	0.008
Nickel	--	--	0.3
Phosphate	82	1.3	--
Potassium	1,890	500	--
Silica	--	--	14.9
Sodium	1,375	160	748
Sulfate	1,280	--	<0.01
Zinc	67	--	18.8

Note: -- means "not measured"

Source: EPA (Ref. 9).

TABLE 4.4. POTENTIAL ORGANIC CHEMICALS IN WASTE LIQUIDS

Organic Chemicals		
Type		Name
Acid	Aliphatic	Acetic acid
Acid	Phenolic	Phenol
Base	Aromatic amine	Aniline
Base	Alkyl amide	Formamide
Neutral-polar	Alcohol	Methanol
Neutral-polar	Aldehyde	Butyraldehyde
Neutral-polar	Alkyl halide	Chloroform
Neutral-polar	Ketone	Acetone
Neutral-polar	Glycol	Ethylene glycol
Neutral-nonpolar	Alkane	Heptane
Neutral-nonpolar	Aromatic	Benzene
Neutral-nonpolar	Alkyl benzene	Xylene
Neutral-nonpolar	Mixed alkane	Paraffin oil
Water		

Source: EPA (Ref. 9).

TABLE 4.5. AVERAGE DATA FROM SEVEN DIFFERENT INDUSTRIAL LEACHATES

Constituent	Highest Average Concentration (mg/l)[1]	Concentration Rank[2]
Chromium	0.87	17
Iron	164	1
Arsenic	120	3
Mercury	0.040	26
Cobalt	1.22	15
Copper	1.62	14
Nickel	4.02	8
Manganese	89.4	4
Cadmium	125.5	2
Zinc	3.95	9
Selenium	<0.005	30
PCBs	4.05	7
Methylene chloride	11.8	5
Trichlorofluoroethane	0.14	24
1,1-Dichloroethene	0.05	25
1,1-Dichloroethane	0.70	19
Trans-1,2-Dichloroethene	4.65	6
Chloroform	1.91	11
1,2-Dichloroethane	2.12	10
1,2,1-Trichloroethane	0.24	23
Carbon Tetrachloride	0.039	27
Bromodifluoromethane	0.039	27
1,2-Dichloropropane	0.035	29
Trans-1,3-Dichloropropene	0.050	25
Trichloroethene	1.69	13
Cis-1,3-dichloropropene	0.050	25
1,1,2-Trichloroethane	0.050	25
Benzene	1.06	16
Bromoform	0.038	28
Tetrachloroethene	0.60	20
1,1,2,2-Tetrachloroethane	0.75	18
Toluene	1.73	12
Chlorobenzene	0.33	22
Ethylbenzene	0.48	21
Chlorodibromomethane	<0.001	31

NOTES:

1. Highest average concentration is equal to the greatest concentration value reported for the constituent in any of the seven leachates.
2. Concentration rank of 1 means that the highest average concentration of the given constituent is greater than all other highest average concentrations for other constituents detected.

Source: TRW (Ref. 8).

Examination of the tables shows that some inorganic constituents appearing in high concentrations in the reported leachates are iron, calcium, manganese, cadmium, and arsenic; and that the organic constituents identified in high concentrations are acetic acid, methylene chloride, butyric and propionic acids, dichloroethanes, and chloroform. The organic compounds by themselves are good solvents. Obviously each leachate or waste liquid requires chemical analysis for identification of constituents and characterization. Permeation of liquid is said to have occurred when liquid penetrates the polymer. Whether or not liquid is transported through the membrane depends upon the concentration gradients and other factors in the environment.

4.3 NATURE OF CHEMICAL COMPATIBILITY

Within the FML industry, a "compatible" FML material is generally accepted as one that is resistant to chemical attack as judged by its changes in physical properties after exposure to the waste. However, the chemical attack can take place via several routes, all of which can be mutually interactive. They are:

- Chemical reaction,
- Hydrolysis,
- Solvation/plasticization,
- Environmental response (heat, light, ozone, bio-organism, etc.).

In chemical reaction, direct reaction of the reagent with active sites on the polymer chain is involved. Subsequent molecular chain scission, addition, or crosslinking may occur. The rate of reaction depends on the polymer and the reagent and, like all chemical reactions, it is greatly affected by temperature and reagent concentration.

Hydrolysis is concerned with the sensitivity and reactivity of a polymer with water. It is a special case of chemical reaction. Since leachate is mostly water, and water cannot be avoided in the environment, it is required that the polymer be little affected by water. However, in the presence of water, the deleterious effects of other chemicals such as ionic species and partially soluble organic substances are frequently accelerated, especially when the polymer sorbs water. Water can act as a carrier for harmful ingredients to the polymer.

Solvation is similar to chemical attack in that an aggressive material, usually an organic solvent, can enter the polymer, dissociate bonds, and reduce it to a solution. Plasticization is a lower degree of solvation in which the solvent is not fully miscible with the polymer. Swelling of a polymer in a solvent is a form of solvation since the chemical has entered the polymer's physical structure.

The environment can affect compatibility by accelerating liquid penetration or chemical reaction. Heat, ultraviolet light, ozone, and some organisms can cause an attack on polymers. Environmental stress-cracking is a form of chemical attack in which a chemical that does not appreciably attack or dissolve a polymer in the unstressed state will cause catastrophic failure when the polymer is stressed in its presence. Initiation and propagation of cracks and crazes occur prior to physical failure. Stress-cracking phenomena are a potential problem with some varieties of HDPE and other semi-crystalline polymers.

Mechanistic aspects of polymer reactions and degradation are discussed in detail in texts concerning the subject and need not be discussed here (Refs. 10-12).

It is important to remember that although we are dealing specifically with tests for chemical compatibility, FML chemical resistance is influenced or altered by all factors capable of producing polymer degradation:

Thermal,
Mechanical,
Photo and high-energy radiation,
Biological,
Enzymatic, and
Chemical.

The definition of compatibility or chemical resistance is therefore open to interpretation. In the present case, we will deal with direct reagent or liquid chemical interaction with FML.

4.4 COMPATIBILITY MEASUREMENT FEATURES

A variety of properties has been proposed and used for evaluating membranes. Predicted serviceability has been based on the properties assumed to be indicators of durability. Of primary interest is resistance to chemicals in waste liquids. Chemical resistance or compatibility is usually based on physical test data gathered after exposure to a chemical or leachate. Physical test data may include tensile properties such as tensile strength, yield strength, elongation at break, elongation at yield, and some others. These are one-dimensional, short-term, simple tests that are easily accomplished in the laboratory, and that are preferred by the industry as service indicators. Additional physical tests may include tear and puncture resistance of the membrane, hardness, and, in the case of a reinforced membrane, ply adhesion tests.

Current methods that have evolved within the rubber and plastics industries are primarily static in principle, that is, samples of FML are not stressed during exposure. Specimens are simply contacted with

chemical under set conditions. Then physical measurements are made. In a waste site, some degree of stress or load must be expected on the FML, even though the FML does not function as a load-bearing material in the engineering sense. Site stresses are not easily identified, nor quantified. Methods in which the specimen is dynamically stressed during the test may provide relevant information about compatibility behavior more realistically.

The method of exposing specimens (e.g., immersion vs. one-surface exposure) needs to be considered in testing and presently is a matter of choice. Laminated and reinforced membrane materials require different handling from homogeneous membranes.

Presently the industrial approach for assessing serviceability rests upon measuring selected physical characteristics and upon qualitative observation. Benchmark data are primarily derived from unexposed product, and comparison is made to products exposed to the environment or chemical. From this approach, chemical compatibility data and specifications for membranes have evolved. The underlying measurements and observations are those that are most easily made, not necessarily the most relevant. The benchmark data are product-specific and become the standard for comparison, even though their applicability to real service is not fully demonstrated.

Testing is not generally conducted with an engineering safety factor or reliability factor in mind. Traditionally, in engineering, material strength safety factors range from 2:1 to 10:1, depending upon materials and requirements. In fact, to achieve a degree of safety, fabric-reinforced membrane is recommended by some FML suppliers. However, the argument for reinforced membrane is based more on circumstantial experience than on measured effectiveness, although some polymers, e.g., CSPE, require fabric reinforcement in most applications due to shrinkage and low modulus when warm.

While it is fairly well accepted that a material that exhibits dramatic changes in physical properties upon exposure to a waste is incompatible, reliable criteria for compatibility have not been established. Field data that support the laboratory measurements are being developed (Ref. 13).

Permeation measurements have been urged by A.D. Schwope (Ref. 2) as an additional means of quantifying the barrier effectiveness of FMLs. The ability of a membrane to restrict or permit migration of a reagent from one side of the membrane to the other without physically destroying the membrane requires serious consideration as a feature of the chemical compatibility test protocol. Haxo recently reported on the permeability testing of polymeric membrane lining materials (Ref. 14). August and Tatzky (Ref. 15), as well as J.P. Giroud (Ref. 16) have also done work in permeability measurement.

5.0 PRESENT COMPATIBILITY TESTS

5.1 BACKGROUND

The chemical compatibility of polymeric material is an important issue not only for the FML industry, but also for the bottle, package, and pipe industries, whenever aggressive or corrosive liquids must be contained. Test methods for determining the compatibility or chemical resistance of a material are used to evaluate polymers in all these industries. All of these industry tests have much in common, having evolved from technological testing in the rubber and plastic industries. In its simplest form, the containment material is exposed to a liquid at some fixed condition, and then physical testing is conducted to determine or measure any changes in its properties. Usually such a test is conducted in the absence of stress, although stress-crack measurements may be made for some polymers. Little attempt is made, on a regular basis, to actually measure the transport of a liquid through the membrane as a measure of compatibility. Water, aggressive liquid reagents, and leachate are all used to challenge the barrier material. Whether the barrier material is to be used for an FML, pipe lining, or a bottle, the final compatibility test is always made with the liquid that is to be contained.

A number of literature sources review polymer testing at several technical levels. Characteristics of polymer testing are evident from the literature. It is not our purpose to develop a theory of testing here. The reader is referred to these sources for further background and scientific discussion (Refs. 12, 17-22).

The chemical resistance of polymers is an inherently more complex matter than that of metals, where the standard expression "mils per year attack" is clearly measured and the effect understood. The term "chemically resistant polymer" is necessarily vague, since each word has degrees of meaning. The strength and functionality of a polymer material is altered not only by aggressive chemical attack, but also by time and temperature. Water, frequently termed "the universal solvent," not only plays a role in accelerating the degrading action, but can by itself be an aggressor (Refs. 23, 24). In any case, the purpose of the FML is to prevent the passage of the challenge liquid or constituents of the liquid into the environment in spite of all other circumstances. A measure of how well the barrier functions is based on the chemical compatibility test. The question is:

Does the test adequately measure the requirement?

Most tests, as we shall see, measure chemical resistance indirectly. After exposure to a reagent, some physical property of the material is measured by mechanical means. A stress-strain measurement on the FML is a widely accepted technique for generating

chemical resistance data. Stress-strain and dimensional change measurements are related to polymer structure and behavior.

Although the FML rubbers and elastomers are visco-elastic materials, creep and fatigue measurements are almost never made. Direct chemical analysis by techniques such as infrared spectroscopy, chromatography, or mass spectrographic methods is rarely conducted by FML producers, although polymer producers and research institutions employ such techniques for polymer characterization.

5.2 TEST METHODS

The search for information on compatibility test methods included not only tests strictly intended to determine the chemical resistance of rubber and plastic membranes such as FMLs, but also allied tests (e.g., for pipe, packaging, and film). All types of tests that were relevant to chemical resistance and the transfer of liquids through membranes were considered and reviewed.

Levels of information sources are categorized as follows:

I. International standards and tests;
II. National standards and tests;
III. Industrial standards and tests;
IV. Project tests; and
V. Academic/literature tests.

The organizations, along with the identification code used in the text for which tests are reviewed, are listed below:

International Organization for Standardization Geneva, Switzerland	(ISO)
Deutsches Institut fur Normung Berlin, Germany	(DIN)
British Standards Institution London, England	(BSI)
U.S. Environmental Protection Agency Washington, D. C.	(EPA)
National Sanitation Foundation Ann Arbor, MI	(NSF)
American Society for Testing Materials Philadelphia, PA	(ASTM)
U.S. Military Standards	(MIL)

We used the key words in Table 5.1 in our searches. To adequately conduct the searches for chemical compatibility test methods, we found this extensive list of topic areas required consideration.

TABLE 5.1. KEY WORDS USED IN LITERATURE SEARCH

(Based on Applied Science and Technology Index with Additions)

Absorption
Absorption Apparatus
Admix
Adsorption
Asphalt
Asphalt Lining

Chemical Apparatus
Chemical Compatibility
Chemical Plants, Corrosion
Chemical Plants, Environmental Aspects
Chemical Plants, Waste
Chemical Resistance
Chemical Standards
Chemistry Apparatus
Compatibility
Composite Materials
Composite Materials, Testing
Containers, Testing
Containers, Flexible
Containers, Plastic
Corrosion, Testing
Corrosion Research
Corrosion-resisting Materials
Corrosion-resisting Materials, Testing

Diffusion
Diffusivity

Elastomers
Elastometers

Flexible Containers
Flexible Membranes
Flexible Membrane Liners

Geomembrane
Geotextile

Lagoons, Sewage
Landfills
Leachate
Leaching

Mass Transfer
Membranes

Packaging, Permeability
Packaging, Testing
Permeability
Pipe, Testing
Plastic Film
Plastic Film, Permeability
Plastic Lining
Plastic Sheeting
Plastics, Aging
Plastics, Crazing
Plastics, Failure
Plastics, Specifications
Plastics, Strength
Plastics, Stretching
Plastics, Swelling
Plastics, Testing
Plastics, Wear
Plastics, Laminated
Plastics, Laminated, Failure
Plastics Research
Polymers Testing

Roofing
Roofs, Plastic
Rubber, Aging
Rubber, Strength
Rubber, Swelling
Rubber, Testing
Rubber, Artificial, Aging
Rubber, Artificial, Strength
Rubber, Artificial, Swelling
Rubber, Artificial, Testing
Rubber Goods, Testing
Rubber Lining
Rubber Research
Rubber Tanks
Rubberized Fabrics

Solubility
Sorption

Tests
Testing
Testing, Equipment
Testing, Machines

Waste, Disposal
Waste, Products
Waste, Lagoons

Of the 27 tests reviewed in Section 5.3, nine deal strictly with FML/waste testing. Of the nine, two tests, NSF Standard 54 and EPA Method 9090, are in contention as a standard method. All other tests described methods for polymeric film and sheeting in general.

For all the tests in general, the method of exposure consists of immersion of a test specimen in the challenge liquid, or of exposure of one side to a reagent. Usually the test is conducted without straining the sample. None of the tests has been created for field use, although field-exposed samples obviously can be brought to the laboratory for measurement. All test methods depend upon specified reagents, and some include water as the chemical challenge.

Actual challenge chemicals are suggested for final testing, but no standard waste compositions or leachates have been specified for laboratory screening. The temperature specified is 23°C or service temperature, but elevated temperatures are specified in several cases. The duration of the test is quite variable--from hours to months--and, in some cases, until equilibrium is achieved. Evaluation of compatibility is based primarily on weight and dimension change, as well as upon several mechanical properties. Almost no failure criteria are given in the methods. It is left up to the tester to decide what constitutes a failure or significant alteration in properties. This evaluation depends upon the investigator's or manufacturer's experience and is based upon familiarity with the product.

5.3 COMPARISON OF TEST METHODS

Tables 5.2 to 5.6 compare, in summary fashion, the relevant features of the test methods pertinent to chemical compatibility testing. Aside from the details, a similarity is seen among types of tests. All the tests were developed for compatibility characterization in the laboratory.

Following the tables are detailed descriptions of these tests. They follow in the same order as in Tables 5.2 to 5.6.

Figures 5.1 to 5.13 describe schematically selected tests typical of available test methods. These figures are useful in judging quickly the complexity of tests and flow of work involved.

TABLE 5.2. INTERNATIONAL STANDARDS AND TESTS

TEST AND/OR SOURCE	METHOD	CHALLENGE	TEMPERATURE	DURATION	MEASUREMENTS	FAILURE CRITERIA	COMMENTS	EQUIPMENT *
ISO 175 PLASTICS--EFFECT OF LIQUID CHEMICALS INCLUDING WATER	IMMERSION	CHEMICALS, WATER	50 C 23 C	24 HOUR 1 WEEK 16 WEEK	WEIGHT DIMENSIONS MECHANICAL PROP (NOT SPECIFIED)	NONE GIVEN	MEASUREMENT OF PROPERTIES AFTER IMMERSION AND AFTER IMMERSION FOLLOWED BY DRYING.	1,8,9,10,11
ISO 1817 VULCANIZED RUBBERS--RESISTANCE TO LIQUIDS	ONE-SIDE EXPOSURE OR IMMERSION	CHEMICALS	SERVICE TEMP	TO BE DETERMINED	WEIGHT DIMENSIONS TENSILE STRENGTH ELONGATION MODULUS HARDNESS	NONE GIVEN	DURATION DETERMINED BY WEIGHT STABILIZATION. MEASUREMENTS MADE AFTER IMMERSION AND AFTER IMMERSION FOLLOWED BY DRYING.	1,5,8,9,10, 11,12
DIN 53 521 RUBBER--RESISTANCE TO LIQUIDS, VAPORS, AND GASES	ONE-SIDE EXPOSURE OR IMMERSION	WASTE	SERVICE TEMP	22,70 HOURS 7 DAYS, UNTIL EQUILIBRIUM	WEIGHT DIMENSIONS VOLUME HARDNESS ELONGATION TENSILE STRENGTH	NONE GIVEN	DURATION DETERMINED BY WEIGHT STABILIZATION.	1,5,8,9,10, 11,12
DIN 53 532 PERMEABILITY OF ELASTOMERS TO LIQUID FUEL	ONE-SIDE EXPOSURE	CHEMICALS	23 C	EQUILIBRIUM	WEIGHT	NONE GIVEN	DURATION DETERMINED BY WEIGHT STABILIZATION.	4,9
BS 4618 CHEMICAL RESISTANCE OF PLASTICS TO LIQUIDS	IMMERSION	WASTE	23 C, MAX. SERVICE TEMP	EQUILIBRIUM	DIMENSIONS APPEARANCE TENSILE STRENGTH FLEX SHEAR IMPACT	NONE GIVEN	DURATION DETERMINED BY WEIGHT STABILIZATION.	1,8,10,11,15
BS 5173 HOSES--CHEMICAL RESISTANCE	ONE-SIDE EXPOSURE	CHEMICALS	SERVICE TEMP	22,70 HOURS 7 DAYS	TENSILE STRENGTH HARDNESS ELONGATION	NONE GIVEN		5,8,11,12

* SEE LEGEND AT END OF TABLE 5.4

TABLE 5.3. NATIONAL STANDARDS AND TESTS

TEST AND/OR SOURCE	METHOD	CHALLENGE	TEMPERATURE	DURATION	MEASUREMENTS	FAILURE CRITERIA	COMMENTS	EQUIPMENT *
EPA METHOD 9090	ONE-SIDE EXPOSURE	WASTE	50 C	30, 60, 90, 120 DAYS	WEIGHT DIMENSIONS TENSILE STRENGTH TEAR PUNCTURE HARDNESS ELONGATION	REFERRED TO EPA RCRA DOCUMENTS		2,5,7,8,9,10 11,12,13,14
NSF STANDARD 54	IMMERSION IMMERSION UNDER STRAIN	WASTE	50 C 25 C	SHORT TERM- 1,3,7 DAYS LONG TERM-- 1,7,14 DAYS AND 1,2,4 MONTHS	WEIGHT DIMENSIONS APPEARENCE TENSILE STRENGTH TEAR MODULUS	NONE GIVEN	ADDRESSES IMMERSIONS OF FML'S WHILE UNDER STRAIN.	1,8,9,10,11
ASTM D543 RESISTANCE OF PLASTICS TO CHEMICAL REAGENTS	IMMERSION	CHEMICALS	23 C	7 DAY	WEIGHT DIMENSIONS MECHANICAL PROP (NOT SPECIFIED)	NONE GIVEN		1,8,9,10,11
ASTM D814 RUBBER-VAPOR TRANSMISSION	ONE-SIDE EXPOSURE	WASTE	23 C	120,192 HOURS	WEIGHT	NONE GIVEN		6,8.9
ASTM G20 CHEMICAL RESISTANCE OF PIPELINE COATINGS	ONE-SIDE EXPOSURE	WASTE	NOT SPECIFIED	6,12,18 MONTH	NOT SPECIFIED	NONE GIVEN		1,8,10,11
ASTM D471 RUBBER-EFFECT OF LIQUID	IMMERSION	WASTE	SERVICE TEMP	22,46,70,166, 670 HOURS	WEIGHT DIMENSIONS TENSILE STRENGTH ELONGATION HARDNESS TEAR	NONE GIVEN		1,8,9,10,11, 12,13
MIL-T-6396D AIRCRAFT TANKS	ONE-SIDE EXPOSURE	FUEL	23C	3,5,8 DAYS	WEIGHT	NONE GIVEN		1,5.9

* SEE LEGEND AT END OF TABLE 5.6

TABLE 5.4. INDUSTRIAL STANDARDS AND TESTS

TEST AND/OR SOURCE	METHOD	CHALLENGE	TEMPERATURE	DURATION	MEASUREMENTS	FAILURE CRITERIA	COMMENTS	EQUIPMENT *
SCHLEGEL	IMMERSION	WASTE	70 C	AFTER WEIGHT STABILIZATION (28 DAY MINIMUM)	WEIGHT DIMENSIONS TENSILE STRENGTH ELONGATION	GIVEN	IMMERSION ENDS AFTER WEIGHT STABILIZES. FAILURE CRITERIA GIVEN.	1,8,9,10,11
J.P. STEVENS	ONE-SIDE EXPOSURE IMMERSION	WASTE	160 C 50 C 23 C	1,2,3,4 MONTHS	WEIGHT DIMENSIONS TENSILE STRENGTH TEAR ELONGATION MODULUS SEAM STRENGTH	NONE GIVEN	ONE-SIDE EXPOSURE IS A POND-TYPE TEST DEVELOPED BY STEVENS. THE IMMERSION TEST IS AN ASTM TEST.	1,2,8,9,10, 11,13
GUNDLE	IMMERSION	WASTE	50 C 23 C	7, 30. 60, 90 DAYS	WEIGHT DIMENSIONS TENSILE STRENGTH TEAR ELONGATION	NONE GIVEN	REFERENCE IS MADE TO NSF METHOD.	1,8,9,10,11, 13

* SEE LEGEND AT END OF TABLE 5.6

TABLE 5.5. PROJECT TESTS

TEST AND/OR SOURCE	METHOD	CHALLENGE	TEMPERATURE	DURATION	MEASUREMENTS	FAILURE CRITERIA	COMMENTS	EQUIPMENT +
SIMULATION TEST (HAIO)	ONE-SIDE EXPOSURE IMMERSION	LEACHATE	23 C	12 MONTHS 56 MONTHS	WEIGHT DIMENSIONS TENSILE STRENGTH ELONGATION HARDNESS WATER ABSORPTION PUNCTURE RESISTANCE SPECIFIC GRAVITY ASH VOLATILES EXTRACTABLES	NONE GIVEN	METHOD IS FOR EXPOSING LINERS TO A LEACHATE.	2,5,8,9,10, 11,12,14
POUCH TEST (HAIO)	POUCH OF LINER MATERIAL FILLED WITH WASTE.	LEACHATE, WATER	23 C	552 DAYS (CONTINUING)	WEIGHT pH ELECTRICAL CONDUCTIVITY	NONE GIVEN	EVALUATES THE PERMEABILITY OF LINER.	1,4,9,16,17
TUB TEST (HAIO)	OUTDOOR TUB EXPOSURE	WASTE	AMBIENT	506 DAYS	DIMENSIONS TENSILE STRENGTH TENSILE SET ELONGATION MODULUS PUNCTURE RESISTANCE	NONE GIVEN		2,9,10,11,14
IMMERSION TEST (HAIO)	IMMERSION	WASTE	23 C	15 DAYS 1,2,4 MONTHS 5 YEARS	WEIGHT DIMENSIONS VOLATILES EXTRACTABLES TEAR TENSILE STRENGTH PUNCTURE RESISTANCE HARDNESS ELONGATION	NONE GIVEN		1,8,9,10,11, 12
NSF FML PROJECT	IMMERSION	WASTE	23 C 50 C	SHORT TERM 7 DAYS LONG TERM 1,7,14,28,56 DAYS 24 MONTH	WEIGHT DIMENSIONS TENSILE STRENGTH TEAR APPEARANCE	NONE GIVEN		1,7,8,9,10, 11,13
HARWELL	IMMERSION	WASTE	40 C	4 MONTH	WEIGHT APPEARANCE ELONGATION TENSILE STRENGTH FAILURE-STRESS LOAD EXTENSION HARDNESS	NONE GIVEN		1,8,9,10,11, 12

+ SEE LEGEND AT END OF TABLE 5.6

TABLE 5.6. SELECTED ACADEMIC/LITERATURE TESTS

TEST AND/OR SOURCE	METHOD	CHALLENGE	TEMPERATURE	DURATION	MEASUREMENTS	FAILURE CRITERIA	COMMENTS	EQUIPMENT *
SEQUENTIAL CHEMICAL ABSORPTION, CARPENTER & FISHER, MATERIALS PERFORMANCE	IMMERSION	CHEMICALS	23 C	30, 120 DAYS	WEIGHT DIMENSION HARDNESS	>10% WT GAIN OR LOSS OF WT BY DESORPTION.	MEASUREMENTS TAKEN EVERY 4 TO 7 DAYS.	1,5,8,9,10, 12
FLUID RESISTANCE OF RUBBERS AND ELASTOMERS, MINTER & MEIER, MATERIALS ENGINEERING	IMMERSION	CHEMICALS	26 C 70 C 100 C	1,7,14, 28 DAYS	VOLUME HARDNESS	NONE GIVEN	COLLECTION OF DATA PRESENTED.	1,5,8,10,12
STRESS CRACKING BY CREEP RUPTURE TEST, MODERN PLASTICS	IMMERSION UNDER STRAIN	CHEMICALS	23 C	UNTIL FAILURE	TIME TO FAILURE	NONE GIVEN		2,5,8
CHEMICAL STRESS RELAXATION, OKUDA	IMMERSION, UNDER STRAIN	CHEMICALS	40 C	NOT DEFINED	ELONGATION SPRING TENSION	NONE GIVEN	CHANGE OF PROPERTIES MEASURED AS FUNCTION OF TIME.	2,5,8,10
PROPOSED-ENVIRONMENTAL STRESS CRACKING OF ETHYLENE PLASTICS	IMMERSION UNDER STRAIN	CHEMICALS	75 C	UNTIL FAILURE	TIME TO FAILURE	NONE GIVEN	HELD AT CONSTANT STRESS UNTIL FAILURE.	2,5,8

* LEGEND

1. SIMPLE CONTAINER (JARS, BOTTLES, BEAKERS, DISHES)
2. SPECIAL CONTAINER (TANKS, LARGE VESSELS, UNIQUE CONSTRUCTION)
3. PRESSURE CONTAINER (APPLIED PRESSURE)
4. POUCH
5. FIXTURES (JIGS, MOUNTING AND SUPPORT GUIDES)
6. PERMEATION CELL
7. STIRRER
8. TEMPERATURE REGULATOR
9. BALANCE
10. MICROMETER
11. STRESS-STRAIN APPARATUS
12. HARDNESS INDICATOR
13. TEAR TESTER
14. PUNCTURE TESTER
15. IMPACT TESTER
16. VOLTMETER
17. PH METER

5.3.1 ISO 175 - Plastics - Determination of the Effects of Liquid Chemical including Water (Ref. 25)

Test Description

This is an immersion test in which at least three precut specimens are used for each test. It is conducted at 23°C and 70°C. Samples are conditioned before immersion according to another ISO standard. The test solution is the liquid to which the material will be exposed in actual use, or, if this liquid is unavailable, a list of test liquids is given.

The preferred test durations are:

a) 24 hours for a short test,
b) one week for a standard test, and
c) 16 weeks for a long-duration test.

During the immersion, the liquids are stirred at least once a day. If the test lasts longer than seven days, the liquid is replaced with an equal amount of the original liquid every seven days.

At the end of an immersion period, the samples are removed, rinsed with a product that has no effect on them, and blotted dry. Weight and dimension measurements are made on the samples. They are then dried at 50°C for 2 hours and the measurements are made again. This procedure of making a measurement, drying the samples, and repeating is also used for the mechanical testing of the samples. Separate specimens of the sample material are used in each part. No specific mechanical test types or methods are listed.

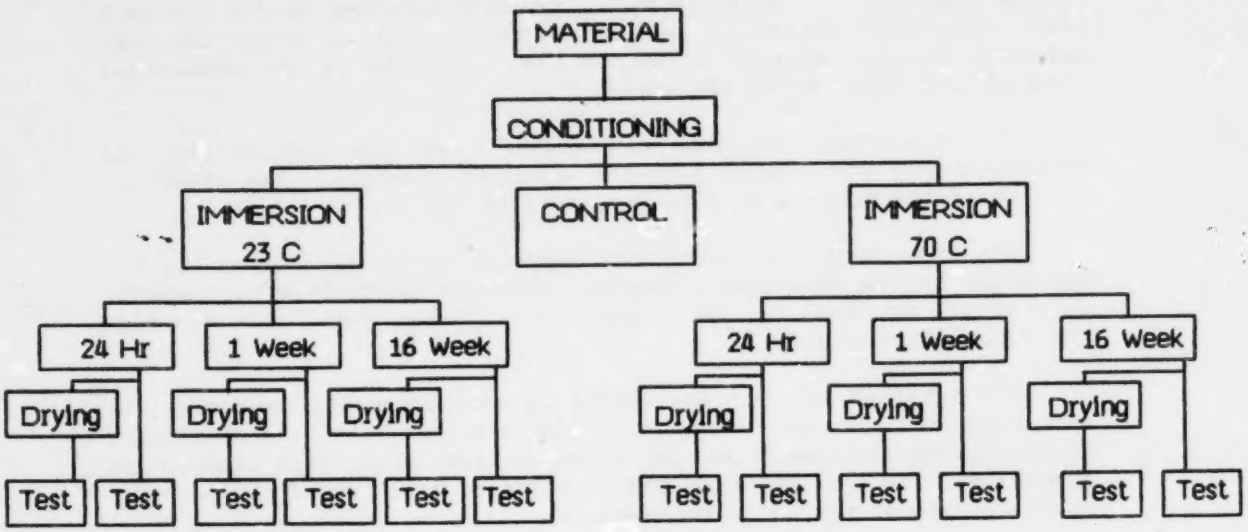
Scope

This method specifies a method of exposing rubber specimens to liquid chemicals and is followed by measuring the changes in properties resulting from such exposure.

Referenced in Test

ISO 62, ISO 291, ISO 3205.

ISO 175



EVALUATIONS

Weight

Dimensions

Mechanical (not specified)

Figure 5.1

5.3.2 ISO 1817 - Vulcanized Rubbers - Resistance to Liquids (Ref. 26)

Test Description

This test can be conducted by immersion or by one-sided exposure of the specimen. At least three precut specimens are needed for each test. The test liquid is preferably the liquid to which the test material will be exposed in actual use. If this is not available, several test liquids are suggested.

The time-lapse between vulcanization of the rubber and the testing should not exceed 3 months whenever possible. The preconditioning of the samples is done according to ISO 471.

The temperature at which the exposure is to take place is not specified in the standard. However, whenever possible, the temperature should be equal to, or slightly more severe than, that at which the rubber will be used.

The duration of the test should be established by measuring the penetration of the test liquid into the rubber. This is accomplished by monitoring the change in the volume or dimensions with time during immersion. The test duration should continue well beyond the point of stabilization of the measured quantity.

The physical properties determined after immersion are tensile strength, elongation at break, modulus, and hardness.

The same physical properties of a second set are measured after drying the immersed samples under a pressure of 20 kPa at 40°C to a constant mass.

Scope

Evaluates the resistance of vulcanized rubbers to liquids.

Referenced in Test

ISO/R 37, ISO 48, ISO/R 471.

5.3.3 DIN 53 521 - Determination of Resistance to Liquids, Vapors, and Gases (Ref. 27)

Test Description

This test is run either as a one-sided exposure or as an immersion test using at least three precut specimens for each test. The exposure vessels are not specified in the method other than that they not allow vapor to escape. The test liquid is preferably the actual exposure liquid. However, if this is not available, a series of the test fluids listed can be used. The test temperature should be slightly higher than the service temperature, but it must be known that no degradation processes take place at this higher temperature. The volume of the test fluid is at least 15 times the volume of the exposed membrane samples.

The exposure time should be greater than the time required for the materials to reach equilibrium. Suggested are the following exposure periods or multiples thereof: 22 hours, 70 hours, or 7 days. The control is an unexposed sample.

The properties measured include weight, dimensions, volume, hardness, tensile strength, and elongation at break.

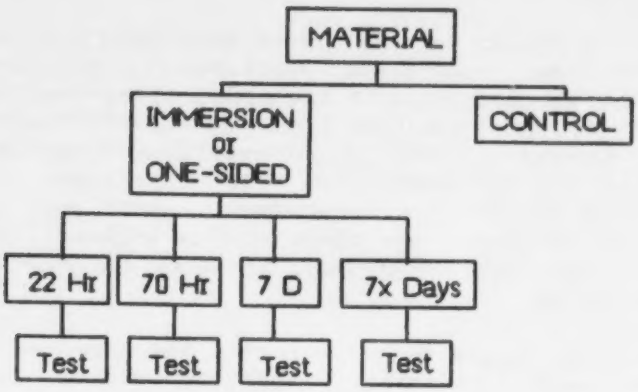
Scope

Evaluates the resistance of rubbers and elastomers to various liquids, vapors, and gases.

Referenced in Test

DIN 50 014, 51 604 Part 1, 53 502, 53 504, 53 505, 53 519 Part 1, 53 538 Parts 1, 2, 53 598 Part 1.

DIN 53-521



EVALUATIONS

Weight

Dimensions

Volume

Hardness

Tensile Strength

Elongation

Figure 5.2

5.3.4 DIN 53 532 - Permeability of Elastomer Sheeting to Liquid Fuels (Ref. 28)

Test Description

For this test, a precut specimen of the elastomer is clamped over the top of a test vessel which has been filled with a particular fuel. The device is allowed to equilibrate for 24 hours in the upright position. The device is then weighed and inverted to begin the exposure period. Weight measurements are taken every seven days until the weight loss becomes a constant for four successive measurements. The permeability can then be calculated by a formula presented in the method. Three specimens are required for each test.

Scope

This method is used to determine comparable permeabilities of materials for a given fuel.

Referenced in Test

DIN 1301, 50 014, 53 521.

5.3.5 BS 4618 - Chemical Resistance of Plastics to Liquids (Ref. 29)

Test Description

This test can be run either as an immersion or as a one-sided exposure. At least two precut specimens are required for each test. As a first phase, the plastic materials are exposed to a variety of the chemicals listed. Materials that show good resistance to chemicals of interest are selected for exposure testing to the actual waste liquid.

The exposure vessels for this test are not specified. Only one type of material is allowed per exposure vessel. The temperatures for the exposures are 23°C and the manufacturer's maximum recommended service temperature.

The exposure is terminated when only small changes occur in the material with time. If the material is still changing significantly after 16 weeks, it should be classified as non-resistant. The controls for this method are samples exposed to the atmosphere for the duration of the immersions.

The properties measured are dimensions, appearance, tensile, flexural, and shear or impact strength as appropriate for the material.

Scope

Evaluates the resistance of plastics to liquid chemicals.

Referenced in Test

ISO/R 175 and BS 3502, 3978, 148, 1911, 2782.

BS 4618

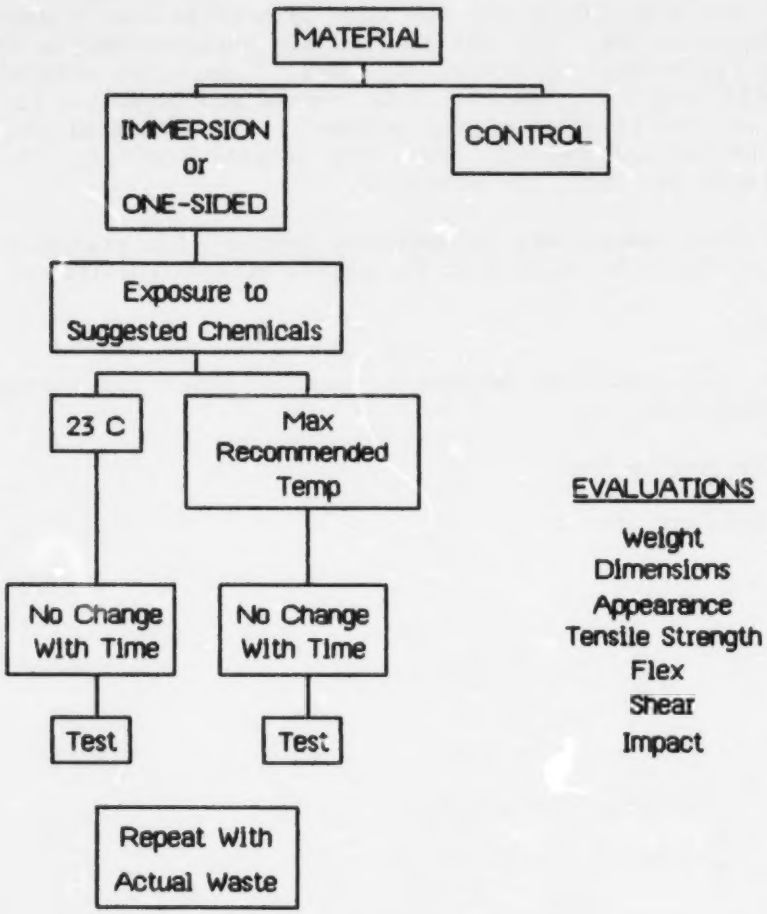


Figure 5.3

5.3.6 BS 5173 - Hoses - Chemical Resistance Tests (Ref. 30)

Test Description

This test is conducted by bending a length of tubing into the shape of a large U and filling it with the actual exposure liquid. The ends are then capped and the hose is held in the U position for the exposure period. The duration of the exposure period is chosen from the following: 22 hours, 70 hours, 7 days, or multiples of 7 days. After exposure, the liquid is removed and inspected for changes in color or the presence of any sediment. The lining of the hose is then inspected and physical tests for tensile strength, elongation, and hardness, and such, are conducted.

The test temperature is selected from a list presented in the method and should be related to the actual service conditions.

Scope

This test specifies methods of testing hoses for resistance to liquid chemicals.

Referenced in Test

BS 903 Part A16.

5.3.7 EPA Method 9090 - Compatibility Test for Wastes and Membrane Liners (Ref. 31) (A Proposed Standard)

Test Description

This test method calls for a one-side exposure. Jigs provide a method of holding slabs of sheeting in tanks such that only the surface of the liner material that would face the waste in actual use contacts the sample of waste fluid.

The exposure tank is equipped with a means of maintaining the solution at a temperature of 50 ±2°C and for preventing evaporation of the solution. The waste liquid is stirred continuously.

Samples of the liner materials are removed at 30, 60, 90, and 120 days of exposure, cut out, and tested for tear and puncture resistance, elongation at break, tensile strength, and hardness. Three specimens are required for each test. Changes in weight and dimensions are also measured. All of these measurements are compared to those of unexposed samples.

Scope

Determination of the effects of liquid wastes on liner materials.

Referenced in Test

FTMS 101B, Method 2065--Tensile and Tear Tests (Table 5.7).

NOTE: Since completion of this study, a revised Method 9090 has been issued by the EPA which includes testing at both room temperature and 50°C. By immersion, both sides of the liner material are now exposed to the chemical environment, although the option remains to expose one side in specific cases. Other features of the revised test are unchanged.

The revised Method 9090 has been proposed for incorporation into the EPA publication "Test Methods for Evaluating Solid Waste" (SW-846), which will become part of the revised RCRA Subtitle C regulations. (See Federal Register, Vol. 49, No. 191, Monday, October 1, 1984, p 38786.)

TABLE 5.7. TENSILE AND TEAR TESTING PROTOCOLS

Property to be Tested	Type of Compound and Construction[a]			
	Flexible Membrane Liners without Fabric Reinforcement			Fabric-reinforced Flexible Membrane Liners
	XL	TP	CX	FR
Tensile Properties				
Method	ASTM D412	ASTM D638	ASTM D638	ASTM D751, Mtd B (modified[b])
Type of Specimen	Dumbbell	Dumbbell	Dumbbell	1 in. x 5 in. strip and 2 in. jaw separation
Speed of Test	20 ipm	20 ipm	2 ipm	12 ipm
Values to be Reported	Tensile strength, psi Elongation at break, % Tensile strength set after break, % Stress at 100, 200, and 300% elongation, psi	Tensile strength, psi Elongation at break, % Tensile strength set after break, % Stress at 100, 200, and 300% elongation, psi	Tensile strength at yield, psi Elongation at yield, % Tensile strength at break, psi Elongation at break, % Tensile strength set after break, % Stress at 100, 200, and 300% elongation, psi	Tensile strength at fabric break, ppi Elongation at fabric break, % Tensile strength at ultimate break, ppi Elongation at ultimate break, % Tensile strength set after break, % Stress at 100, 200, and 300% elongation, ppi
Tear Resistance				
Method	ASTM D624	ASTM D1004 (modified[c])	ASTM D1004	(d)
Type of Specimen	Die C	Die C[e]	Die C[e]	
Speed of Test	20 ipm	20 ipm	2 ipm	

[a] XL = crosslinked or vulcanized; TP = thermoplastic; CX = crystalline; FR = fabric-reinforced.

[b] Specimen = 1 in. x 5 in. strip; initial jaw separation = 2 in.

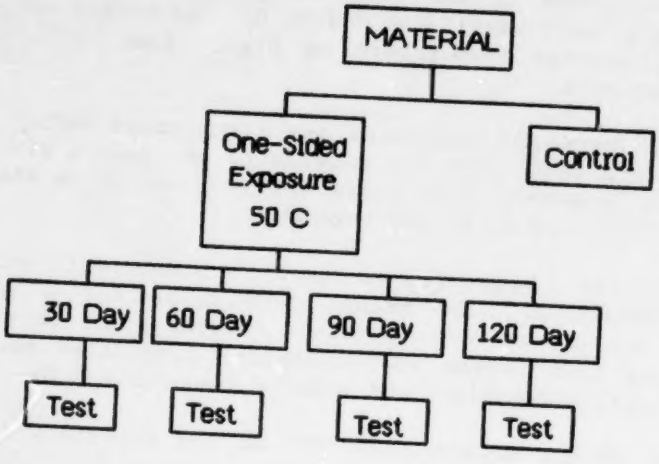
[c] Crosshead speed = 20 ipm.

[d] No tear resistance is recommended for fabric-reinforced sheetings in the immersion study.

[e] Die C from ASTM D624.

Source: EPA Method 9090 (Ref. 31).

METHOD 9090



EVALUATIONS

Weight
Dimensions
Tear Strength
Tensile Strength
Puncture Resistance
Elongation
Hardness

Figure 5.4

5.3.8 NSF Standard 54 Flexible Membrane Liners (Ref. 32)

Test Description

Precut samples are completely immersed in the test fluid. Specific immersion equipment is not specified. Two test temperatures are suggested: room temperature (23°C) and 50°C. The method suggests that if the actual service temperature is higher than 50°C, that temperature should be used.

There are short-term and long-term tests suggested using this method. The short-term test calls for samples to be removed after 1, 3, and 7 days. The long-term test calls for samples to be removed after 1, 7, and 14 days, and 1, 2, and 4 months.

After exposure, the samples are examined for changes in weight, dimensions, and appearance. The method also calls for a gentle solvent rinse with acetone or other suitable solvent prior to examination. Samples are then tested for mechanical properties such as tensile strength, tear, elongation, and modulus (see Table 5.8).

Also addressed is the effect of strain on the materials during the immersion. Samples are shaped into a loop, immersed for 7 days or 4 months, and then removed and inspected visually for deterioration.

Scope

This standard deals with providing a uniform method of specifying and testing of FMLs. Only Appendix D of NSF Standard 54 deals with the estimation of performance in chemical environments and is reviewed here.

Referenced in Test

See Table 5.8.

TABLE 5.8. TEST METHODS

Crosslinked Elastomers Unsupported - ASTM D412 (Method A)

- Breaking strength, pounds/inch width
- Elongation at break, percent
- Modulus at 100 percent elongation

Thermoplastics - Unsupported

Materials without a yield point - ASTM D882

- Breaking strength, pounds/inch width
- Elongation at break point, percent
- Modulus at 100 percent elongation, pounds/inch width

Materials with a yield point - ASTM D683

- Breaking strength at yield and break, pounds/inch width
- Elongation at yield and break, percent
- Modulus of elasticity, pounds/inch width
- Tear strength, unsupported FML - ASTM D1004

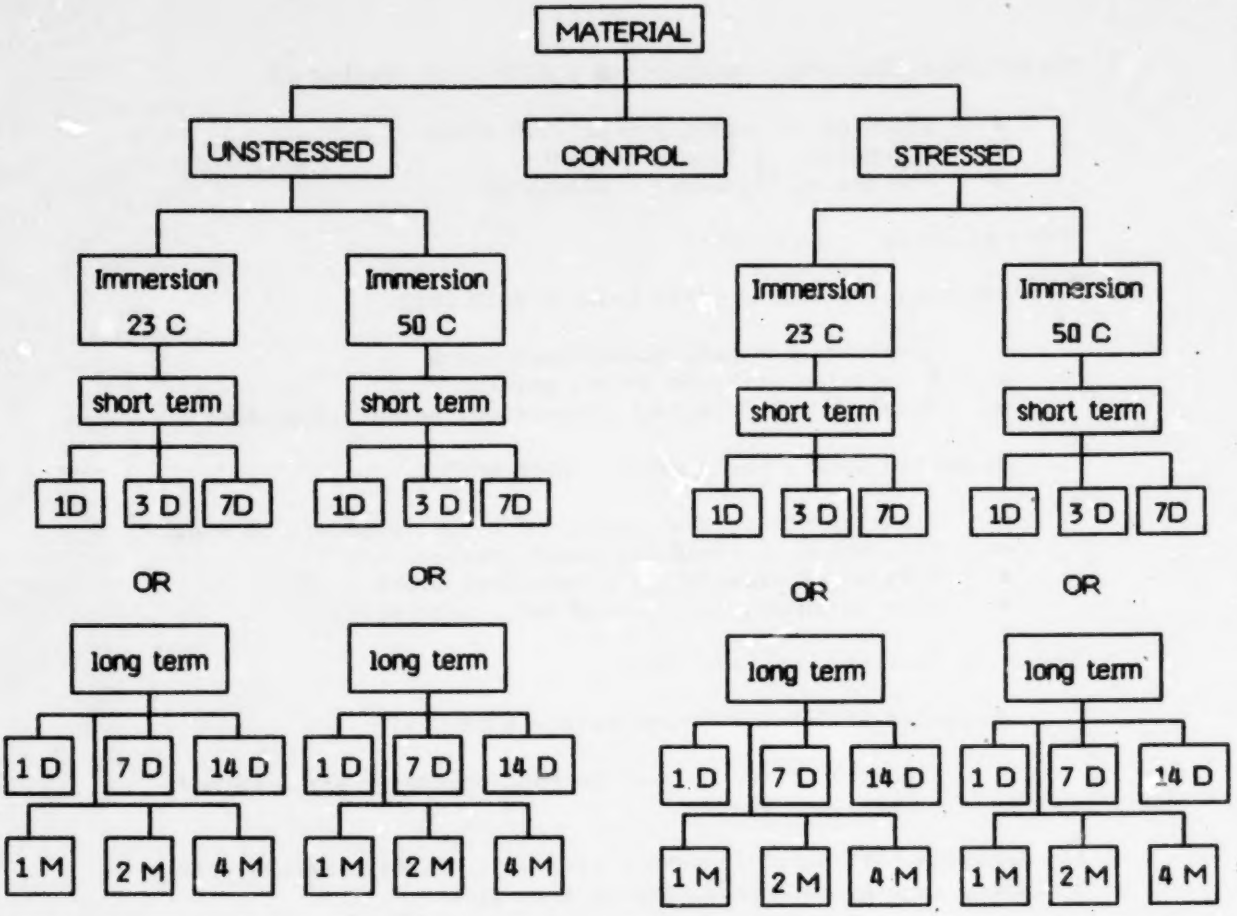
Supported Flexible Membrane Liner

Supported finished FML materials - ASTM D751

- Breaking strength, grab method, pounds or strip method, pounds/inch width

Ply Adhesion, All-supported FML - ASTM D413, Machine Method, Strip Specimen, Type A, 180° peel, pounds/inch width

NSF STANDARD 54



EVALUATIONS

Weight
Elongation
Tensile Strength
Tear Resistance
Modulus

Figure 5.5

5.3.9 ASTM D543 (78) - Resistance of Plastics to Chemical Reagents (Ref. 33)

Test Description

Precut test specimens, at least three replicates per type test, are immersed in a chemical liquid at 23°C for 7 days. Water is an optional liquid. For other than room temperature, 50°C and 70°C are suggested. Other immersion times may be used (suggested are 1 and 3 days and increments of 4 weeks). Changes in visual appearance, weight, and dimension are determined. Changes in tensile properties (no methods specified) are determined as well. Fifty standard reagents are specified.

Scope

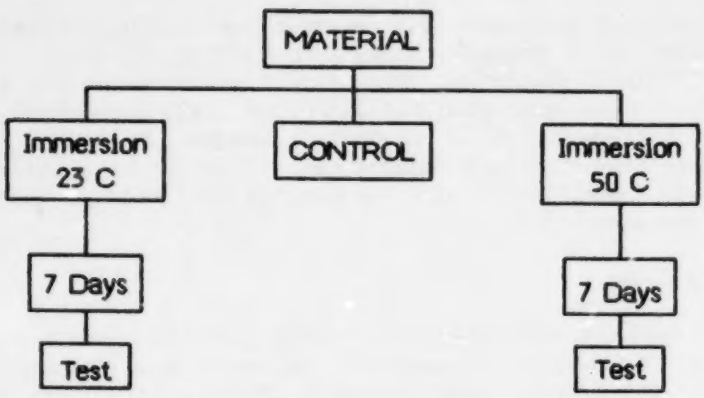
This test is suitable for testing sheet polymeric materials for resistance to chemical reagents. Reported are changes in weight, dimension, appearance, and strength times. Provision is made for various exposure times and exposures at elevated temperatures.

This is a laboratory test and may not be related to real conditions. Choices of reagents, temperatures, times, and measured properties are arbitrary. The test is the basis of standardized comparative testing. No criteria for failure or serviceability are given.

Referenced in Test

ASTM D618, D833, D1898.

ASTM D543



EVALUATIONS

Weight
Dimensions
Mechanical (not specified)

Figure 5.6

5.3.10 ASTM D814 - Rubber Property - Vapor Transmission of Volatile Liquids (Ref. 34)

Test Description

For this test, a disk of rubber material is clamped over the mouth of a jar which contains the test liquid. The jar is weighed and inverted and then placed in a suitable rack and allowed to stand for 24 hours. The jar is then reweighed and returned to the rack. The jar is reweighed again after 120 hours and 192 hours from the initial inversion. The vapor transmission rate is calculated from any loss in weight. At least three specimens are required for this test. This test is run at room temperature.

Scope

This method is designed to measure the relative difference in vapor transmission rates of various rubbers.

Referenced in Test

ASTM D3767.

5.3.11 ASTM G20 - Chemical Resistance of Pipeline Coatings (Ref. 35)

Test Description

This is an immersion test designed for pipe coatings. The immersion fluid should contain reagents anticipated in actual service. The immersions are carried out in glass jars with pipe sections capped at both ends as the test samples. The length of the immersions are 6, 12, and 18 months. The temperatures of the immersions are not specified in the method. The properties to be measured before and after exposure are not specified.

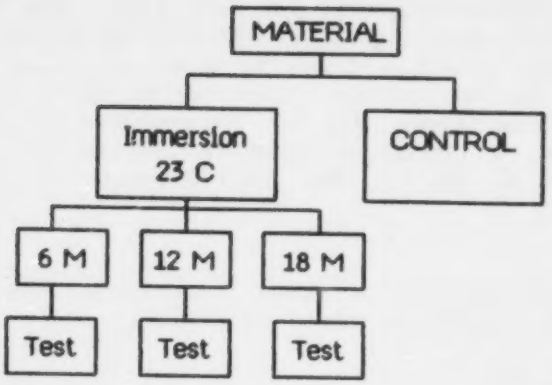
Scope

The test evaluates the resistance of exterior pipe-coating materials to various reagents.

Referenced in Test

ASTM G8, G17, D543.

ASTM G20



EVALUATIONS

Not Specified

Figure 5.7

5.3.12 ASTM D471 - Rubber Property - Effect of Liquid (Ref. 36)

Test Description

For this ASTM test, precut test specimens are completely immersed in the test fluid--three specimens for each test. The test fluid is preferably the actual waste liquid, but if this is unavailable, alternate fluids selected from a list can be used. A series of different test temperatures may be selected from tables found in the method description. The temperature used for the exposure should be the one that comes closest to the actual service temperature. Recommended immersion times are 22, 46, 70, 166, and 670 hours.

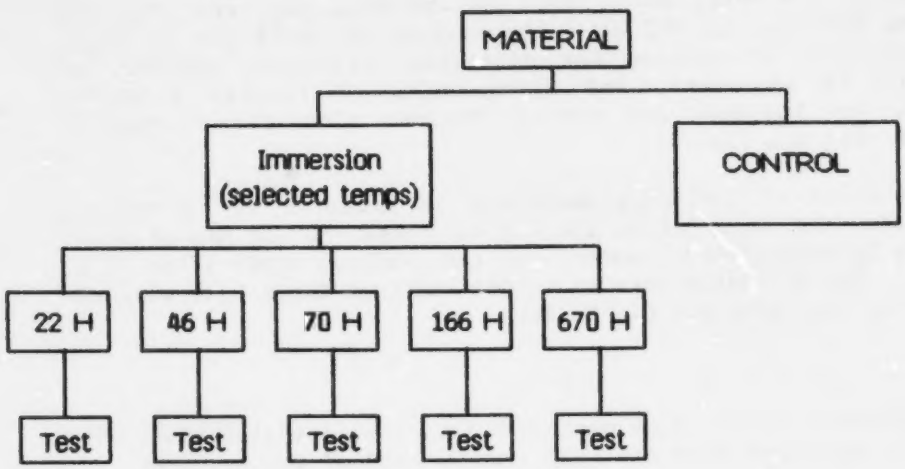
Scope

This method is designed to yield comparative data on which to judge the effect of liquids on rubber materials.

Referenced in Test

ASTM D412, D1193, D1415, D2240, D3182, D3183.

ASTM D471



EVALUATIONS

Weight
Dimensions
Tensile Strength
Elongation
Hardness

Figure 5.8

5.3.13 MIL-T-6396D Aircraft Tanks - Fuel, Oil, Water-alcohol, Coolant (Ref. 37)

Test Description

Samples of the tank, inner liner ply and outer ply, are subjected to relative humidity of 95% at a temperature of 160°F for 30 days. Visual inspection of samples for corrosion, cracking, warping, or delamination is conducted, and measurements of tensile strength, elongation, and hardness are taken. Maximum allowable changes in these properties are listed.

Permeability of the tank materials is measured by affixing a sample of the tank material to the top of a half-filled cup of fuel. This device is weighed and inverted and then weighed again after 3, 5, and 8 days. The diffusion rate is calculated from the weight measurements made on the fifth and eighth days.

Scope

This document covers many phases of fuel tank specification. The two sections described here are only a small part of the document.

5.3.14 Schlegel Test for HDPE (Ref. 38)

Test Description

This is an immersion test that is conducted at 70°C. No special equipment is required. A sample of the waste fluid is used as the test reagent. Specimens are precut.

Initial measurements of weight and dimensions are made, and then the samples are immersed in the waste fluid for a total of 28 days. Every 7 days the samples are removed, wiped dry, and weighed. After 28 days, if the sample weights have stabilized, the immersions are discontinued and property testing is begun. If the weights have not stabilized, the immersions are continued until the weights become constant.

When the immersions are concluded, tensile strength and elongation measurements are made on the exposed samples and compared to measurements made on unexposed liner samples.

A test specimen is determined to have passed the test if:

a. the weight change is not greater than ±3%,
b. the change in tensile properties is no greater than ±10%, and
c. the liner seams are not significantly weaker than the liner sections.

Scope

Determines chemical resistance.

Referenced in Test

ASTM D618, D638.

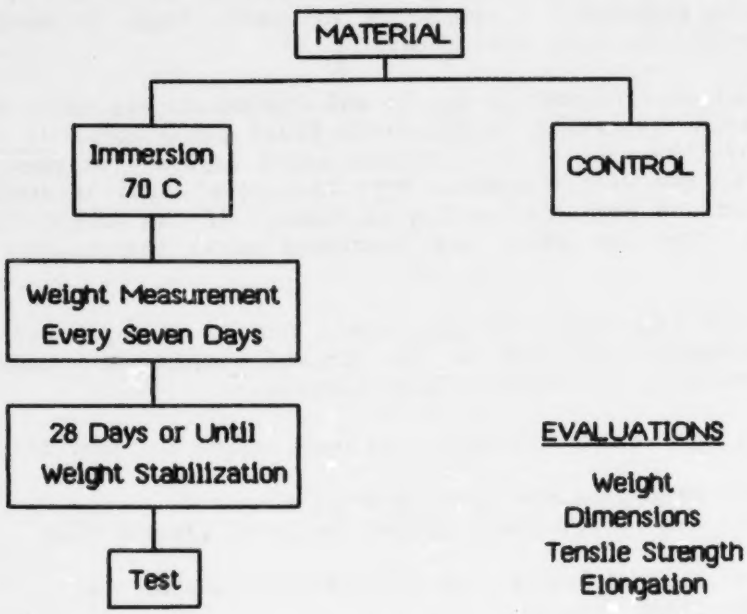

SCHLEGEL
MATERIAL
Immersion
70 C
CONTROL
Weight Measurement
Every Seven Days
28 Days or Until
Weight Stabilization
Test
EVALUATIONS
Weight
Dimensions
Tensile Strength
Elongation

Figure 5.9

5.3.15 J.P. Stevens Test (Ref. 39)

Test Description

The finished lining is reinforced with polyester. To measure change in physical properties and percent weight, unsupported CSPE is laboratory-milled to 0.015 inch from which test specimens are taken. Test specimens are immersed for up to four months in the actual waste liquid at 23°C and 50°C. Physical property changes are compared to the original. The percent change by weight is conducted at 23°C and 50°C, and results are plotted to establish rate of absorption to equilibrium.

A closed cell ponding test allows the waste liquid to be exposed to one side of the finished reinforced lining at elevated temperatures up to 160°F. A glass cover is sealed over the waste liquid to prevent evaporation. The test allows for natural phase separation of the waste.

An open air ponding test holds up to 2 inches of the waste liquid which, under sunlamps, produces a surface temperature of 160°F and an effluent temperature of 135°F. The waste is allowed to evaporate leaving only the solids. The lining can be resaturated with more waste. Physical properties and seam strength tests can be made upon completion of the test.

Scope

Evaluation of lining material in actual waste liquid.

Referenced in Test

ASTM D412, D1004, D471.

5.3.16 Gundle Test (Ref. 40)

Test Description

This is an immersion test conducted at 23°C and 50°C. Precut samples are prepared for tensile and shear testing, according to ASTM D638. Specimens are also prepared as 1x4-in. strips to be used for visual inspection, weight measurement, and dimensional change determination.

Samples are removed from liquid contact after 7, 30, 60, and 90 days for evaluation. Strength testing is done at each time interval. Weight and dimension measurements are conducted on the strips which are then returned to the solutions.

The test solution for this evaluation is the supplied waste fluid.

Scope

This test evaluates the liner material by immersion in waste solution.

Referenced in Test

ASTM D543, D638.

5.3.17 Simulation Test (Haxo) (Refs. 9, 41)

Test Description

Exposure unit: a 2-foot diameter steel pipe, 10 feet in height, is placed on an epoxy-coated concrete base and lined with polyethylene. The liner is sealed into the base with epoxy. A layer of sand is then placed on top of the liner. Approximately 1 cubic yard of ground refuse is compacted above the sand to yield a density of 1240 pounds per cubic yard at 30 percent water content. The refuse is covered with 2 feet of soil and 4 inches of crushed rock. One cell for each liner for each time period is required.

Tap water is added at the rate of 25 inches per year. Leachate is maintained at a 1-foot head above the liner by continually draining the pipe just above the liner.

In addition, specimens of the liner are also buried in the sand above the primary liner and thus are totally immersed.

When the samples are removed, they are subjected to a range of physical tests normally performed on rubber and plastic materials.

Scope

This method is suitable for exposing any type of liner materials to simulated municipal solid wastes.

5.3.18 Pouch Test (Haxo) (Refs. 9, 41)

Test Description

A small pouch is made with the liner material. The pouch is filled with the waste material or any test fluid, and then sealed and immersed in de-ionized water. The permeability of the membranes to water and to pollutants is determined by observing the change in weight of the bags and the measurements of the pH and electrical conductivity of the de-ionized water. Because of osmosis, water should enter the pouch, and ions and dissolved constituents should leave the bag.

Scope

This test is designed to evaluate the permeabilities of the liner materials to liquid wastes and water.

5.3.19 Tub Test (Haxo) (Ref. 9)

Test Description

Plywood tubs with sides sloping outward at a 1:2 slope are constructed. The dimensions are 14x9 inches at the base and 25x20 inches at the top, creating a depth of approximately 11 inches.

Liner specimens are draped over the tubs and folded to fit the inside contours of the tubs. Excess material is allowed to hang freely over the edges of the tubs. The tubs are then filled about three quarters full with wastes. The liquid level is allowed to fluctuate about 4 inches.

The tubs are kept outdoors during the exposure period. Visual inspections of the liner and the liquid level are conducted at regular intervals. The tubs are covered during rainy weather. The tubs are also located such that half of them are in the sun and half are in the shade.

After exposure the liners are tested for a variety of physical properties.

Scope

This test exposes the liner to conditions that simulate actual conditions.

5.3.20 Immersion Test (Haxo) (Refs. 9, 41)

Test Description

Samples of liner materials are immersed in a tank containing the waste mixture. If the mixture is expected to separate, samples should be hung at different levels. After exposure, the samples are removed, cut, and tested for changes in weight, dimensions, tensile strength, puncture resistance, tear resistance, and elongation; one sample for each immersion time or exposure condition is required.

Scope

To assess the compatibility of liner materials with specific wastes.

Referenced in Test

ASTM D297, D412, D624, D638, D1004, D2240, D3421.

FTMS 101B, Method 2065, Puncture Resistance and Elongation Test (1/8-inch Radius Probe Method)

Matrecon Test Method 1, Procedure for Determination of Volatiles on Unexposed Membrane Liner Materials

Matrecon Test Method 2, Procedure for Determination of the Extractable Content of Unexposed Membrane Lining Materials

5.3.21 NSF's FML Project (Ref. 42)

Test Description

For this NSF project, short-term screening tests and long-term exposure tests are conducted. The short-term screening consists of a 7-day immersion in various organic compounds at three concentration levels. The concentration level that shows a noticeable effect on the liner material without destroying it is used as the highest concentration for the long-term testing. Two dilutions of that concentration level are also used. Distilled water serves as the zero concentration for both sets of tests. The liner materials are evaluated in the short-term tests by appearance, weight, and dimensional changes. These tests are run only at room temperature.

The long-term exposure test is run as an immersion test. The two temperatures used are 50°C and room temperature (23°C). Immersed samples are removed from the exposure tanks for testing after 1, 7, 14, 28, and 56 days, and after 24 months. The properties measured include weight, dimensions, tensile strength, and tear strength. Changes in appearance are noted. Properties are measured according to ASTM standards wherever possible. Stirring of the solutions is not specified. Fifteen precut samples per material, exposure period, and temperature gradient are required.

Scope

The test evaluates the resistance of several commonly used liner materials to specific chemicals at several conditions.

Referenced in Test

ASTM D1593, D751, D882, D1004, D412, D624, D638.

NSF's FML PROJECT

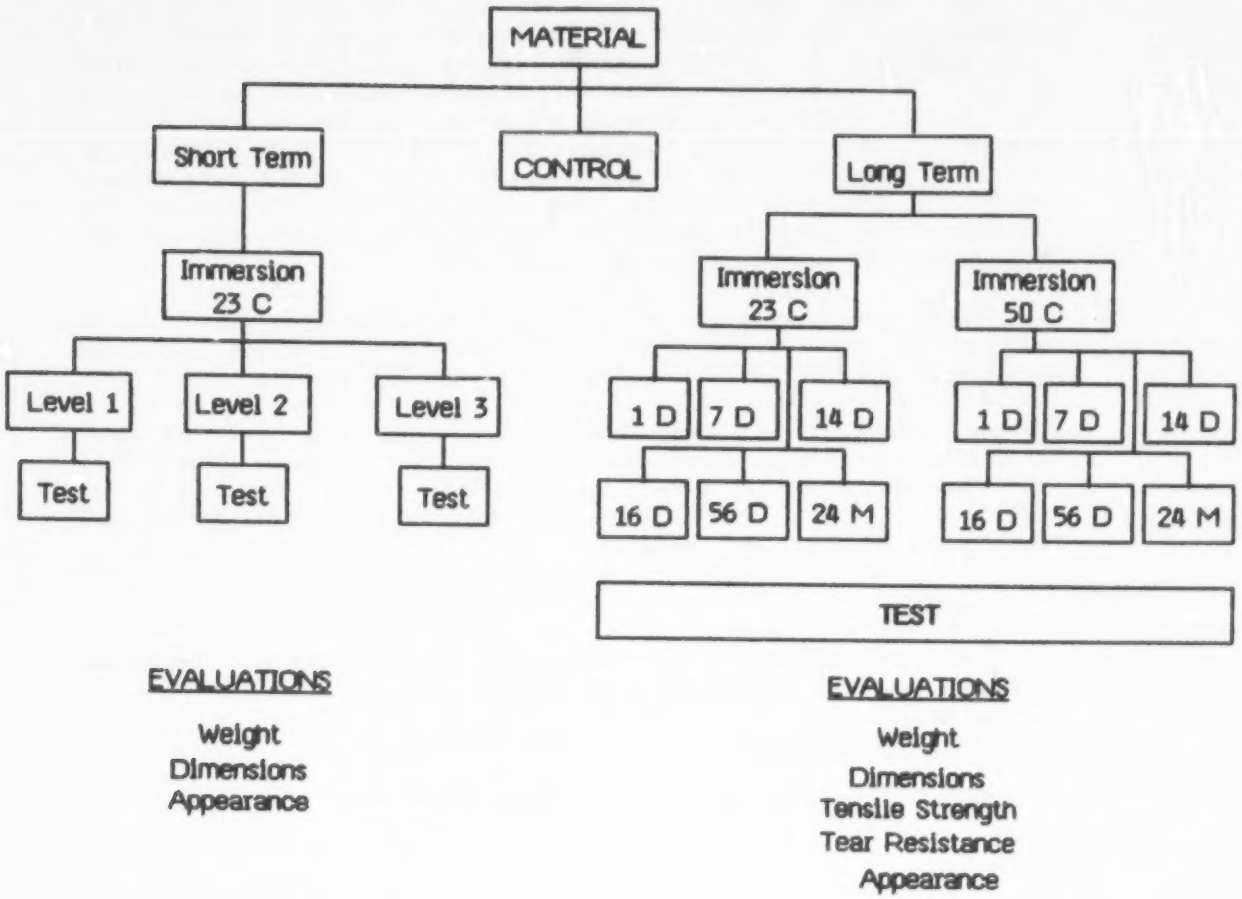


Figure 5.10

5.3.22 Harwell Assessment of Two HDPE Landfill Liners by Application of an Accelerated Test (Ref. 43)

Test Description

The exposure tanks for this test are stainless-steel pressure vessels 0.35 meter in diameter and 0.76 meter high. The temperature in the vessels is maintained at 40°C (representing the upper limit of temperature encountered in an active landfill site in the U.K.). The pressure in the vessel is maintained at 20 psi with nitrogen which prevents oxidation of the leachate.

The composition of the leachate was designed to represent a particularly aggressive version of that from domestic refuse. The composition of the leachate used is given in the source.

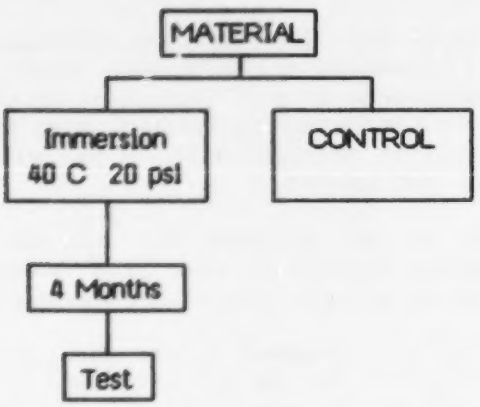
The liner samples are attached to the roof of the vessel above the level of the leachate. To the submerged end of the samples are attached weights (276 g) to maintain the material under tension throughout the test.

After 4 months, the liner samples are removed and evaluated for changes in appearance, weight, elongation, tensile strength, failure stress, load-extension behavior, and hardness.

Scope

This test evaluates liner materials against simulated landfill leachate under accelerated conditions.

HARWELL REPORT



EVALUATIONS

Weight
Elongation
Appearance
Tensile Strength
Tear Resistance
Failure Stress
Load Extension
Hardness

Figure 5.11

5.3.23 Sequential Chemical Absorption Techniques for Evaluating Elastomers (Ref. 44)

Test Description

This test is a modification of ASTM D471. Modifications include: (1) use of a thinner sheet stock for testing to accelerate the approach to equilibrium, and (2) increase in exposure periods from one long period to at least four shorter ones to permit extrapolation of the data to infinite time. The exposures are complete immersions and the properties measured are weight, dimensions, and visual observations. Hardness measurements are made only at the end of the final period. The test period may vary from 4 to 10 days with a total elapsed time of 120 days.

With an integration of Fick's second law of diffusion and the data obtained from the testing, the weight gain at infinite time and the diffusion coefficient for the material are calculated. If this weight gain is greater than 10%, the material is eliminated from further testing.

Desorption of the successful materials, ones with less than 10% weight gain, is then conducted in open jars at room temperature until the weight stabilizes. Any material that shows a loss in weight is considered unacceptable since it indicates leaching of the elastomer.

A preferred material is one that passes both of these tests and does not lose more than 10 durometer points (hardness) over the duration of the testing.

Scope

Evaluates chemical resistance of elastomers by determining maximum absorption values and diffusion and permeation properties.

Referenced in Test

ASTM D471.

5.3.24 Guide to Fluid Resistance of Rubber and Elastomers (Ref. 45)

Test Description

The test method described is an immersion test in which the specimens are exposed in 4-ounce jars. The exposures are carried out at three temperatures: 26°C, 70°C, and 100°C. The materials are tested against water, transformer oil, ASTM oil No. 3, and a chlorinated, aromatic dielectric fluid. The properties measured include hardness and volume changes. Measurements are taken after 1, 7, 14, and 28 days. These tests are conducted with natural rubber and 17 elastomers.

Scope

Evaluates chemical resistance of rubbers and elastomers (compilation of hardness and volume change data for 18 materials versus 4 fluids).

Referenced in Test

ASTM D471.

5.3.25 Environmental Stress-cracking by Creep Rupture Tests (Ref. 46)

Test Description

In this test, the environmental stress-crack resistance is measured in terms of the loss of strength of the material. The test specimen is mounted by clamping one end to the bottom of a cylinder and the other to a lever arm with weights on the arm to create the tension on the sample. The cylinder is filled with a chemical, and the time to rupture the specimen is measured as a function of the applied stress. The test can also be run at elevated temperatures.

Scope

This method measures the stress-crack resistance of plastics in chemical environments.

Referenced in Test

ASTM D1693, D2990.

5.3.26 Chemical Stress Relaxation Test (Ref. 47)

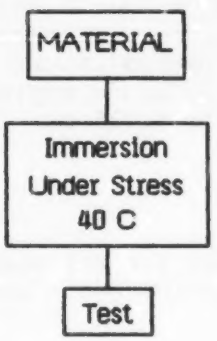
Test Description

In this test, materials are exposed to liquid chemicals while under stressed conditions. The material is held under tension by fixing one end to the bottom of the exposure tank and the other to a spring. A selected load is then put on the spring. The chemical is added to the tank and held at a constant temperature (room temperature, or 40°C) for the duration of the test. The elongation of the test specimen is held constant during the test, while the amount of spring tension required to hold it there is recorded with time. A stress ratio is calculated and plotted against time to develop stress relaxation curves for the material.

Scope

The use of stress-relaxation for evaluating the chemical resistance of plastics is described.

CHEMICAL-STRESS RELAXATION



EVALUATIONS

Elongation

Spring Tension

Figure 5.12

5.3.27 A New Method for Determining Environmental Stress-crack Resistance of Ethylene-based Plastics (Ref. 48)

Test Description

In this test, materials are bent around a metallic form having a specified radius of curvature. A constant load is then applied to the material by attaching weights to a cord that is attached to the material over a series of pulleys. The material is held in this manner in a tank containing the stress-cracking agent, a 10% solution of nonylphenoxy poly(ethyleneoxy)ethanol in distilled water. The temperature of the test is 75°C, the applied stress is 5 MPa. Nominal sample thickness is 0.100 to 0.125 cm. The test is terminated when the material fails.

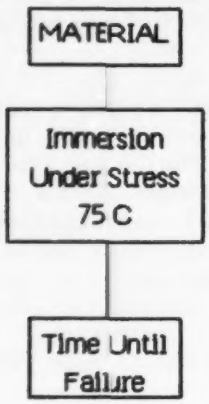
Scope

This method is designed for the determination of environmental stress-crack resistance of polyethylene-based plastics.

Referenced in Test

ASTM D1693, D2552.

ENVIRONMENTAL STRESS-CRACKING OF ETHYLENE PLASTICS



EVALUATIONS

Time under stress until failure

Appearance

Figure 5.13

5.4 TEST STATE-OF-THE-ART

A general feature of the tests described in the previous section is commonality of approach. This is evidenced by the cross-referencing of key tests (e.g., ASTM D543, ASTM 471, ISO 175, ISO 1817, DIN 53 521, BS 4618). The approach appears to be based on assimilating test methods for rubber sheet and plastic sheet into a test useful for FMLs. It is worth noting that a "caveat" is generally found in each method, warning that the test may not correspond with the service condition and that data are only comparative in nature. Absolute values or criteria for compatibility are not established in the tests.

At the present time, two specific test methods for evaluating FML performance in a chemical environment have evolved--NSF Standard No. 54 and EPA Method 9090. The latter was designed specifically as a compatibility test for membrane liners in the presence of waste. In the NSF standard, chemical compatibility testing is described in Appendix D of the document which deals with other aspects of FML specifications. The NSF method is a voluntary approach to industry standardization; the EPA method may be promulgated as a national standard having regulatory authority. The NSF test is primarily short-term, although provision is made for longer testing.

Both tests rely on measurement of similar physical property changes. Criteria for failure are not specified by the NSF test, nor the EPA test, although the EPA test refers to RCRA guidance documents for evaluation of data. The NSF document deals with stress-cracking phenomena in a separate test; the EPA test does not at all. Both employ similar test temperatures (23°C and 50°C). Importantly, in Method 9090 samples are subjected to one-side exposure; in NSF 54, individual test specimens are immersed in a reagent. Both recommend FML contact with actual waste liquid or leachate.

Other FML industry test methods depend primarily upon immersion in fluid, although one-side contact in a simulated ponding arrangement (heated dish or tub) is sometimes used. Attempts are made to simulate real experience, but the pro's and con's of the exposure methods are still being debated. Again, physical and mechanical features are monitored for change. Information on industry test results is scarce because results tend to be proprietary. Industry does retain its own concept as to what constitutes failure in products. Tests are static in nature and single property measurements (e.g., tensile strength, elongation, hardness, and tear strength) suffice for characterization.

Manufacturers and users of polyethylene products are interested in stress-cracking. For these products, this property is tested by stressing a test specimen in a solution of a surfactant. Current work at the Polymer Science and Standards Division, National Bureau of Standards, is being devoted to the investigation of stress-cracking of

ethylene-based plastics in the presence of liquid stress-cracking agents (Ref. 48). A review of stress-crack testing is given by Titow (Ref. 49). Methods discussed are free-bend methods, imposed-curvature methods, three-point bending, plain-strip-specimen, holed-strip-specimen, biaxial methods and others. Choices in stress-crack testing appear just as complex as those in the physical tests described for compatibility.

EPA, NSF, or industry tests have not included permeation measurement in compatibility testing, although much research has been and is underway which should lead to the measurement of this property. If the waste barrier is to be maintained, then passage (via a permeation diffusion mechanism) of liquid through the membrane in the absence of holes should not be permitted.

In summary, the tests reviewed exhibit the following characteristic features:

- Test by immersion or one-side exposure;
- Test without stress, except for the polyethylene-type plastics, which are a special case;
- Test at ambient temperature and some elevated temperature;
- Test with varying time (short to long), or to equilibrium;
- Test in laboratory;
- Test with reagents or waste;
- Measurement of mechanical properties and evaluation of appearance as the preferred indicators of compatibility.

No one test satisfies all needs for a chemical compatibility test, and test protocols and experimental details are subjects for discussion and re-evaluation.

5.5 CONSENSUS MEETING

As part of the need to elucidate and understand compatibility test methods and FML requirements, a meeting with experts in FML technology and applications was held at Arthur D. Little, Inc., Cambridge, Massachusetts, in January 1984. In attendance were several manufacturers' representatives, an independent FML researcher, EPA representatives, and members of the Arthur D. Little staff. This meeting provided an opportunity to discuss the current test methodology and recommendations for improvements in tests for FML compatibility. The discussions identified areas of concern, issues, and means for resolution. This meeting was most useful in bringing to light practical aspects of testing not dealt with in the published test methods discussed in prior sections. Because they provide valuable insight, the Minutes of that meeting are reproduced in Section 5.6.

5.6 MINUTES OF MEETING ON COMPATIBILITY TEST METHODS AND FML REQUIREMENTS

5.6.1 Exposure Conditions

Immersion vs. One-side Exposure

Industry representatives were in favor of immersion testing, but split on the question of whether or not supported materials should be tested with or without the scrim. One argued that if you want to measure the change in strength of the liner, you should leave the scrim in place. Others suggested that to evaluate a change in the material by measuring strength, scrim should not be included since its strength overshadows any change in the material. Ply adhesion between scrim and the barrier material is a concern. Everyone agreed that if scrim were included, the edges should be sealed to prevent wicking of the waste into the liner.

One EPA representative was in favor of one-side exposures since this would accommodate composite liner materials (multi-layered) where chemical resistance was not required for both sides of the liner. Several people responded that composite liners were not practical for the near future because of the technical problems associated with them.

In testing, the rate of chemical attack would be much faster in an immersion test; therefore, any adverse effects would be seen more clearly in a shorter exposure time.

Duration

Most parties agreed that a three-level testing protocol was a sound approach. The three levels are outlined below:

Level	Duration	Temperature	Waste Concentration
Short-term	30 days	Room temperature (23°C) and 50°C	Actual and 10 times by evaporation or spiking
Intermediate	Four months	Room temperature (23°C) and 50°C	Actual and 10 times by evaporation or spiking
Long-term	More than four months	Actual service temperature	Actual

The short-term test was considered a good way initially to screen materials for further testing. Samples should be removed after 1, 7, 14, and 30 days for evaluation.

The 4-month, intermediate exposure did not receive much criticism. Everyone recognized the need for a test with a significant exposure period to demonstrate the resistance of the liner material. Removal of samples after 7, 14, 30, 60, 90, and 120 days for testing was suggested, along with replacing of the waste fluid every 30 days.

Long-term data are important and are urgently needed, both from laboratory and field tests. A field lifetime test was strongly suggested as a means of monitoring the liner during its actual service life. Attaching coupons of the liner material to a frame and placing the frame on the bottom of the pond was mentioned as one method of conducting this test. Samples could then be removed for testing after several years without cutting out a piece of the liner itself.

Temperature

The use of higher temperature for accelerating the test assumes that any degradation process occurring at the higher temperature is the same as those encountered at the lower temperature. One industry representative objected to the use of elevated temperatures for two reasons: (1) the temperature may kill the active micro-organisms that can attack an FML in the pond in actual use; and (2) the loss of volatile solvents in testing would have to be addressed. Another practical problem mentioned was that it may be too dangerous to expose the solvents to the higher temperature. It was agreed, however, that higher temperatures would accelerate chemical degradation taking place as a result of the exposure to the waste.

Concentration

Everyone believed that the liner material should be tested against the actual waste mixture. Exposure to the pure chemical was rejected, since it does not simulate actual conditions where concentrations are usually very low. Exposure to 10 times the expected actual concentration, on the other hand, was considered a good way to simulate a worst case situation that may develop in a pond. However, it was recognized that concentration of a received waste for the test would not always be feasible.

Phase Separation

If phase separation of the chemical in the waste mixture is expected to occur, samples of the liner should be placed at several levels in the exposure tank to intercept each chemical. All of these samples should be evaluated after the exposure period.

5.6.2 Test Methods

All agreed that just one set of tests for all types of liner materials would not be appropriate. The tests must be proper for the type of material being tested. Three major classifications of materials were suggested. Below, commonly used liner materials are classified and typical tests suggested.

Classification Type

1. Thermoplastic
2. Crosslinked
3. Semi-crystalline

Material	Type	ΔWeight	Tear*	Tensile	Elong.	Modulus	Stress-crack
PVC	1	X	X	X	X	X	
HDPE/LDPE	3	X	X	X	X	X	X
CPE	1	X	X	X	X	X	
CSPE (CSM)	1→2	X	X	X	X	X	
EPDM	2	X	X	X	X	X	
ECO	2	X	X	X	X	X	
Neoprene (CR)	2	X	X	X	X	X	
Butyl (IIR)	2	X	X	X	X	X	

*Not with fabric reinforcement

In addition, it was suggested that dimensions and hardness also be checked on all materials since these properties are easily measured.

Static vs. Dynamic

After some discussion, it was agreed that results produced by dynamic testing would be interesting. However, this type of testing of materials is not understood well enough in relation to compatibility at this time. Static testing, on the other hand, was considered adequate to measure changes in the materials caused by the exposures. It was suggested that a research study be conducted to determine the usefulness of dynamic testing (e.g., rheology, creep,

stress relaxation). It appears that testing methodology is related to convenience, cost, and experience.

Controls

Considerable discussion was given to this subject. Several people were of the opinion that untreated material should serve as the test control, while others suggested that immersion in de-ionized water, or even tap water, would be more appropriate. Immersion in de-ionized water had more support than any other method, but there was no consensus. It was also suggested that samples be treated in water prior to any subsequent exposure or test. FML material should be retained for reference in a cool, dry, anaerobic environment.

Permeation

Several industry representatives felt that contamination in the field caused by permeation is insignificant or is not experienced. In cases of leakage in ponds, the industry says it has always been able to trace the leak back to breaks or tears in the liner. Others suggested that permeation was a real problem. However, they did not believe they knew enough about the subject to evaluate it. Most believed that further work in this area should be conducted.

Other Comments

Specimens for physical testing should not be cut from the product sample before exposure to the waste fluid. Samples of the liner materials should be cut out of coupons after the exposure period.

Some samples of the exposed liner materials should be dried in an oven prior to testing to simulate material which is at the water line in a pond and which may dry if the level drops.

5.6.3 Cost Profile

Cost of conducting compatibility tests was not discussed in depth because of insufficient information. EPA (Washington) claims that a current study being conducted using Method 9090 on five materials and one waste will cost $15,000 including equipment. The cost for the construction of the exposure units alone was about $6,000.

The industry representatives thought a figure of $20,000 to $25,000 for five materials and one waste was a realistic cost. They pointed out that only one type of liner material should be run in each exposure unit. A multiple of tanks would be required for a large number of samples, or samples of different types. They also expect that the cost of storing and maintaining all of the exposure units they would need would also be significant. Because of the requirements of Method 9090, a large number of samples would be required.

Sample numbers would compound rapidly for multiple combinations of membranes and wastes for a given supplier required to conduct tests. A laboratory toxic waste-handling and disposal problem would then become a real issue. Additional regulations for health, safety, and environmental requirements would have to be observed.

5.7 TEST EQUIPMENT AND COST CONSIDERATIONS

Besides the technical evaluation of the test methods, equipment requirements and cost should be considered. Basic equipment needs for the tests are outlined in Tables 5.2-5.6. Specific equipment needs vary depending upon the scope of the test, but common items are evident. For example, a container to hold the membrane sample in contact with liquid, means of controlling conditions like temperature, and apparatus, or tools for making measurements will all be required. The degree of complexity of individual tests stipulates the sophistication of equipment needed. For example, the exposure system can vary from glass jars containing liner materials and the wastes placed in an oven, to self-contained units or tanks for waste and liner which are provided with temperature control and condensers to recycle the vapors. Most test methods specify the parameters of the exposure, but not the specific equipment type. Separate units are required for each material and each waste. Any container used must be resistant to potentially corrosive wastes. Means for handling and storing the exposure units during the exposure period are a practical matter. Proper ventilation, for example, should be provided since many wastes contain volatile constituents and may be toxic.

The size of containers may be relatively small (0.5-1 liter jars) or quite large (4-100 liter tanks). Construction of special sample jigs and vessels is required in some tests, especially those where one-side immersion is involved.

Tools to measure dimension, a balance to measure weight change, and possibly a microscope to look at the surface characteristics of membranes, are basic equipment for measurement and observation.

Also indispensable is stress-strain apparatus to measure tensile strength, moduli, elongation, and other mechanical properties of a membrane. A stress-strain apparatus, depending upon model and features, can be priced from $15,000 to more than $60,000 if process control is included. Except in a few instances where equipment specifications or designated apparatus are included in the test method, choices are left to the tester. In reality, the test methods provide a protocol around which the actual test is fashioned. Involved in the scope are types of membranes to be tested, quantity of material, number of test specimens, numbers of waste liquid or chemicals, required measurements, time of testing, and temperature conditions.

Reliable cost information for conducting a compatibility test is unavailable at this time. In the absence of a generally accepted or specified method, independent testing laboratories consulted were reluctant to set a test cost. They were all quick to point out that the cost would depend upon the choice of method, selection of liner and wastes (or chemicals), and specified information needs. Presently, chemical compatibility testing of FML does not seem to be a routine practice for testing laboratories, and therefore they have had little experience with selecting, applying, and costing these test methods. FML manufacturers' test costs are, of course, proprietary, as is private contractors' information. As experience with FML testing in the industry increases, a better understanding of costs will evolve. It is premature to expect reliable test cost information at this point.

6.0 GENERAL APPROACH TO COMPATIBILITY TESTING

6.1 THE SCOPE OF TESTING

As pointed out throughout the previous sections of this report, compatibility testing of FML is made complex by a number of factors and interactions. Although apparatus and protocol may differ, current test methods apply simple, familiar technological tests to evaluate those FML materials being used in a demanding, new application. Each of the tests described earlier appears to be useful for screening and evaluation, but all are inadequate for predicting behavior patterns on a truly long-term basis. Evaluation of test results is based on limited experience, mostly from membrane manufacturers.

Figure 6.1, The Compatibility Test Scheme, outlines the complex nature of compatibility testing. It is a summary of those factors to be considered in making up a compatibility test. All the factors described, as well as the interactions outlined, should be considered if chemical compatibility is to be measured in its fullest sense. No one current test takes into account the complete scheme, and it would be impractical to conduct testing on this basis. Yet, all the features shown are related to chemical compatibility and its assessment. Current tests deal with only small segments of the scheme. Whether or not parts of the scheme are equal to the whole (compatibility) is still a matter for resolution. General discussions of polymer testing are found in the literature (Refs. 20, 50, 51). Chemical compatibility represents only one facet of polymer characterization. Features of the scheme are elaborated upon below and some value judgments are made.

6.1.1 FML Material versus Liquid Challenge

Obviously, for chemical compatibility to exist, the liner membrane must be matched to the waste or aggressive liquid. Selection of the liner material results from general chemical resistance guidelines developed by manufacturers. More detailed compatibility tables may be useful for specific combinations of FML and challenge. For instance, CPE membrane is not recommended for use in aromatic hydrocarbons, and PVC is not useful with oxygenated or chlorinated solvents. Ultimately, whether the liner is tested in field or laboratory, the final test must be conducted with the service chemical, waste, or leachate. The fact that most waste and leachate are dilute solutions, may not be homogeneous, or may exhibit phase separation, all pose additional considerations for the tester. Concentrated solutions and dilute solutions of the same active materials can produce different results.

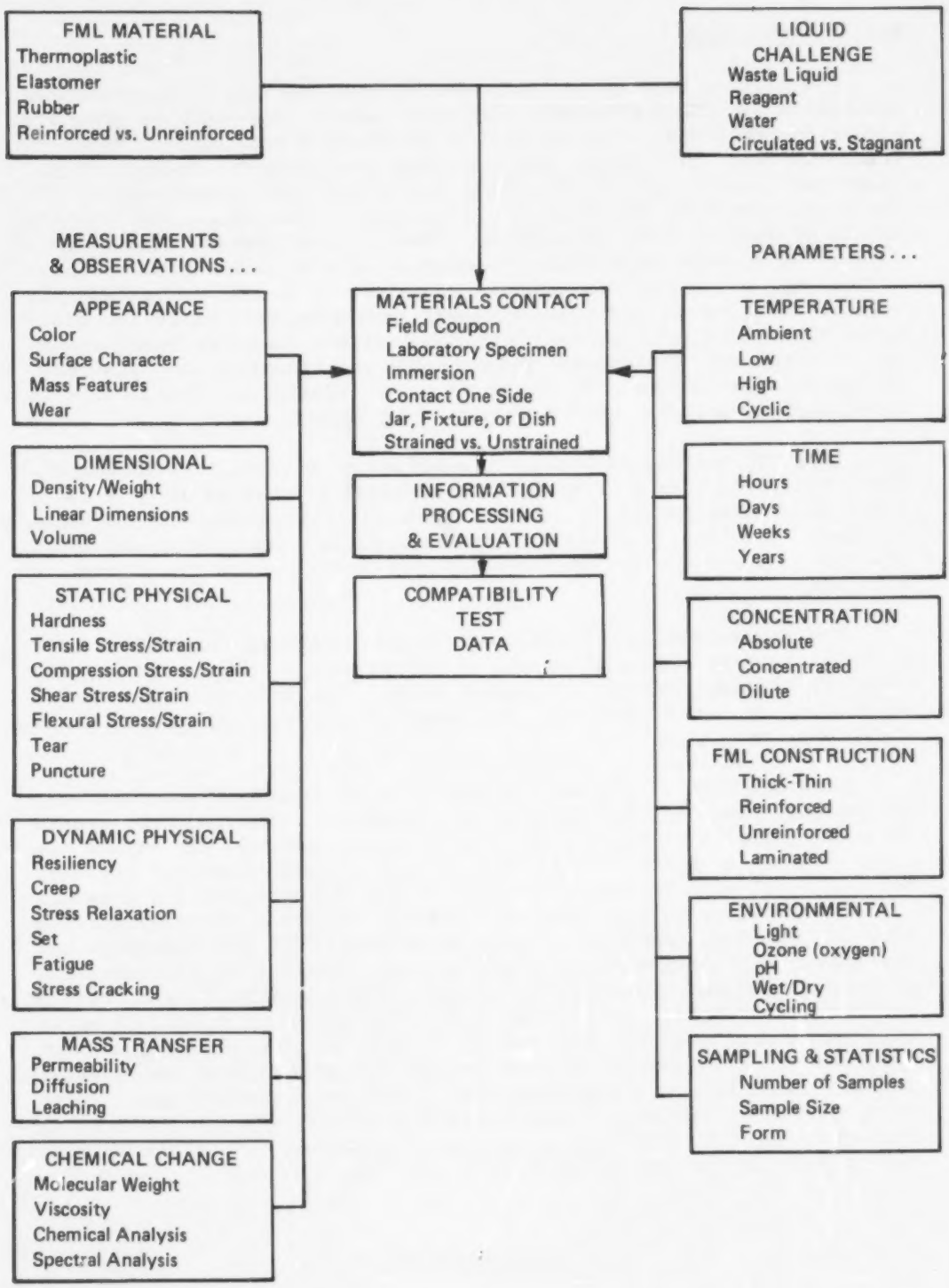


FIGURE 6.1 THE COMPATIBILITY TEST SCHEME

6.1.2 Parameters

The list of parameters represent factors that may be selected and identified in the laboratory, but most likely they will be indeterminate in the field. Temperature in the field may actually fluctuate, concentrations may change, and impinging environmental factors (e.g., light and ozone) may differ from day-to-day and site-to-site. If waste at elevated temperature is present, then obviously testing should be done at this temperature. However, testing chemical compatibility at a high temperature to produce accelerated behavior can be misleading, since high-temperature reactions in polymer materials may bear no relation to reactions at lower temperatures. Above the glass transition point of a polymer its chemical and physical behavior may be altered and deviations from Arrheniun kinetics are possible. Different properties of plastics and elastomer (visco-elastic materials) do not vary with temperature uniformly.

Time for testing is basically a matter of choice. The longer the time, the more closely it approaches expected or desired service life. A series of time points in testing can reveal if equilibrium is being reached. However, practical considerations are important, and tests are not likely to go on indefinitely before decision-making is required.

The concentration of challenge ingredients may vary from 100% material to very dilute solutions of salts, acids, bases, and organic chemicals encountered at the waste site. The role of water as an aggressor, or as a catalyst for chemical aggression, needs to be carefully considered.

Consideration of the FML construction is important and necessary to testing. Reinforced membrane, for instance, should not be tested by immersion without sealing the edges, since chemicals can attack the fiber scrim in a way unlike its action on the membrane surface. Sealing edges with a coating of another polymer selected for chemical resistance is not an easy task, and results are often unreliable. The size of the test sample is important as well. If small samples cut for subsequent machine testing are exposed, swelling or reaction will produce a specimen considerably changed dimensionally from a control or untreated sample. Subjecting larger specimens or sheets of material to liquid contact followed by sample cutting produces a test specimen--in a different swollen or reacted state--that has another set of dimensions. Measured values based on dimension and weight changes will be different between the two techniques. If a sample is too thin, or too thick for apparatus, fixtures, and handling, then alterations in the test protocol are required.

Environmental factors (e.g., light, ozone, site structure, physical features) encountered in the field are not readily duplicated in the laboratory and are generally neglected in compatibility testing, even though they might ultimately affect properties and endurance of the film.

Sampling and statistics are important features of the compatibility test design. Numbers of replicate samples and experimental reproducibility are considerations for generating reliable data. Obtaining representative, homogeneous, and characterized waste liquid for the compatibility test is a major problem. The issue of sample controls or standards for comparison cannot be overlooked, not only in the specific test experiment, but for the manufactured materials as well. It should be remembered that the FMLs are compounded, manufactured products that might vary in composition from batch to batch.

6.1.3 Measurements and Observations

Figure 6.1, under the heading of Measurements and Observations, lists indicators of chemical compatibility. Only a few form the basis for actual chemical compatibility tests now in use. After exposure, appearance is the first clue to compatibility. Mass deformations and color changes are immediate indicators that reaction has taken place. Dimensional changes are also an immediate indicator of change. Swelling of elastomers indicates sorption of a liquid. Swelled membranes can behave differently from unswelled material.

Traditionally, in plastic and rubber testing, the static physical tests led by hardness and tensile stress/strain measurement are used to indicate changes and degradations. The stress/strain properties are related to the molecular makeup of the polymer, so that any attack or alteration in the polymer structural configuration is manifested by stress/strain changes. Most of these physical tests, whether in tension, compression, shear, or bending (a combination of all three) specified for polymers, have been adapted from time-honored methods for metals. In polymer technology, it is assumed that a simple, single test of short-term mechanical nature at an arbitrary combination of time and temperature and in one physical state is useful for evaluating the general performance of plastic material. Although the industrial evaluation relies on these short-term mechanical tests, most experts would agree that such tests only describe partial behavior, and indeed might be unreliable predictors of actual behavior. A special consideration is the fact that in mechanical testing the value and meaning of observed changes are not clear. Is no change in value necessary for compatibility, is 5%, 10%, etc., adequate, or is an equilibrium value sufficient? For some products (e.g., pipes, gaskets, and seals) distinct values are acceptable, but not for liners at this time.

The dynamic physical properties represent a whole group of features, more difficult to evaluate, not routinely conducted, or even suggested, for compatibility assessment. The term dynamic is used to describe the type test in which an elastomer is subjected to cyclic or multi-directional deformation. Stress and strain, or other properties, are monitored during the test. These types of measurement are characteristic of visco-elastic behavior and are time-related. Some would say that dynamic measurements are better related to long-term behavior. Considered under dynamic testing is the stress-cracking phenomena important to the chemical compatibility testing of polyethylene-based polymers. Stress-cracking is dynamic in the sense that the aggression occurs in a flexed condition of the sample.

Mass-transfer properties in membrane films have been neglected in compatibility measurement. However, if one goes back to the model of the barrier membrane, then obviously, any movement of liquid through the membrane via any mechanism negates the effectiveness of the FML. Permeation and diffusion describe a mechanism, in simple form, whereby the leachate or solvent molecules can slip through the tangled molecular chains of a polymer and pass from one place to another. Passage of liquid may occur without chemical degradation, or solvation, in the strict sense. Until recently, much good data of this type have not been readily available for FML. Techniques for measuring permeability and diffusion of small amounts of chemical are more sophisticated than simple immersion and property testing, but are being investigated for this application (Refs. 2, 14, 15, 16).

All methods to this point are indirect measurements for chemical compatibility. Chemical change can be measured directly by analytical techniques that provide insight into the fundamental chemical and physical makeup of the polymer. These techniques might include chemical analysis, spectrographic characterization, molecular weight, viscosity measurement, and the like. Such techniques are preferred by researchers in the field of polymers rather than technologists. They tend to be time-consuming and expensive. Such techniques, most useful for single polymers, become difficult to interpret when applied to mixtures and compounds found in commerce.

6.1.4 Test Details

Many other details in experimental procedures need to be considered. Should the sample be tested by immersion or one-side contact? In jars, cells, tubs, pouches, etc.? Should reagents or leachate be replaced in time throughout the test? What degree of stress and rate of strain should be used in testing? Should exposed samples be examined in the wet or dry state? What ratio of liquid volume to membrane is realistic? Should hydrostatic pressure be applied in testing membranes? What about practical considerations, such as handling specimens and toxic waste materials? The resolution of these questions and others that can arise present real issues. Presently,

they are dealt with on the basis of individual tests, and even at that, in a peripheral manner.

In summary, attention must be given to a variety of factors in testing. They include:

- Compositional nature of test specimen (e.g., type of polymer, type of blend or compound, degree of crosslinking);
- Structure of test specimen (reinforced versus non-reinforced);
- Variability in test sample (lot-to-lot variation and field variation);
- Preparation of sample (size, form, conditioning);
- Choice of liquid challenge (reagent versus leachate);
- Method of exposure (immersion versus one-side; laboratory versus field);
- Conditions of exposure (concentration, temperature and time);
- Mode of stressing sample if any;
- Type of measurement (liquid saturation versus diffusive transfer);
- Parameters measured (chemical versus physical); and
- Variability in results.

6.2 THE CURRENT DILEMMA AND ROUTES TO RESOLUTION

The complexity of FML/waste compatibility testing has been emphasized in this report. Most experts in the field would agree that chemical compatibility testing is no simple matter. An interesting letter (excerpted in Appendix A) received from West German workers states a European opinion and reiterates the complexity.

Yet, to derive useful and reliable data, we are faced with finding and applying a simple test methodology to a subject in which the parameters are not well defined. The establishment of reliability and the life-time prediction of FML in hazardous waste containment is expected to depend upon such evaluations. The several published ASTM and international standards tests for chemical resistance of plastics and rubbers have been considered a step in that direction.

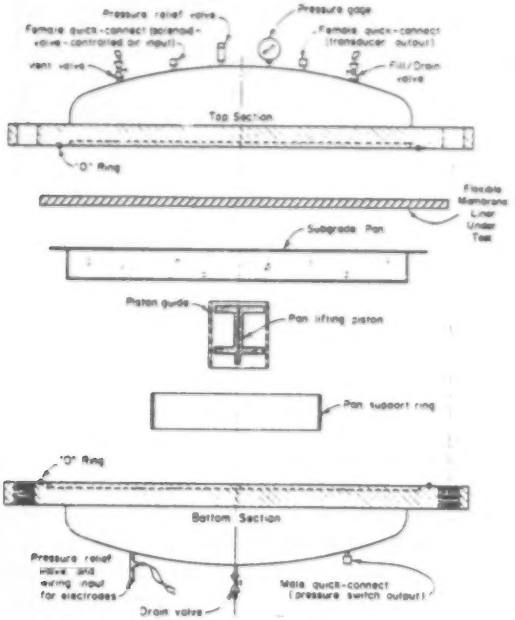
To meet the specific need, NSF 54 and Method 9090 have been created. Of the identified tests, NSF Standard No. 54 and EPA test method 9090 are the only two test methods currently directed to FML. NSF 54 is based on ASTM D543 and ASTM D471 (both described in Section 5.2). A limited number of physical properties (tensile strength, modulus, elongation, weight and dimensions, etc.) are measured after an exposure by a specified technique. It is a short-term test (up to 7 days) conducted at 23°C and 50°C. Two important qualities (low permeability and puncture resistance) for FML were not included for measurement. The NSF standard considers permeability measurements to be meaningless, because FMLs are "impermeable." According to NSF, a good test for puncture resistance does not exist. EPA Method 9090 also depends upon simple physical measurements after exposure. Puncture resistance is measured. A more elaborate test protocol and a greater number of specimens compared to NSF 54 are specified. Test time is longer (up to 120 days). Temperature is elevated to accelerate matters. The NSF test is an immersion technique; the EPA, a one-side exposure. Both procedures specify exposure to a sample of actual waste fluid. Other variations in detail are seen from comparisons of descriptions in Section 5.2. Neither method includes measurement of permeability or stress-cracking, although the NSF method does include a reference to ASTM D1693 for measuring environmental stress-crack resistance.

Are these two methods adequate for evaluating compatibility and predicting life expectancy? Both tests are derivatives of ASTM and international tests. Both derive information with the same inadequacies of the other discussed tests. Physical property data derived from mechanical measurements are the primary indicators for compatibility. Sampling requirements, elaborate protocol, and equipment needs can make Method 9090 expensive to conduct. Although useful for screening and initial evaluations, both produce only an incomplete picture of chemical resistance.

Other tests or alterations in methodology may be advantageous. Carpenter and Fisher (Refs. 44, 52) have worked out relatively simple tests based on their work for evaluating the chemical resistance of elastomers for pipe linings. Their tests are summarized in Section 5.3.23. Carpenter and Fisher have developed an accelerated laboratory test method for rubber linings which establishes maximum absorption values in addition to normal physical changes. The method is a simple modification of ASTM D471 and involves sequential absorption determinations in thin-sheet stock. It is an immersion test. Thin elastomeric sheet stock (1.6 mm) is used for accelerating the test. Accumulated weight change with elapsed time of exposure is plotted. The weight changes are easier to obtain than volume changes. Conditions can be set for a given environment. From this procedure, maximum absorption is determined, and a diffusion coefficient is calculated. Guidelines are given for estimating lining life. Also included is a diaphragm test for permeation measurement.

In assessing pipe liner elastomer, the authors paid attention to the shape of the sorption curve. For pipe corrosion protection, membranes that exhibited any peaks or net weight loss are rejected as lining material. Interestingly enough, their thesis for chemical testing elastomers is based primarily on weight change. Weight change in rubbers has historically been used as a measure of performance. No tensile or other test is required, although hardness may be measured. This sequential chemical absorption technique, if applied to FML and backed by experience, might provide the simplest approach to testing.

An alternate test approach in which the laboratory might be made to simulate the site condition is to adopt the automated hydrostatic flexible membrane test facility developed at the Bureau of Reclamation (Ref. 53). In this case, a large membrane (∿20" dia.) can be tested in the cell containing actual subsoil (Figure 6.2). A challenge liquid under a hydrostatic load would stress the membrane. Temperature and test times could be altered as desired. After exposure, the membrane would be removed and tested for appropriate properties.



Adapted from R.K. Frobel (Ref. 53).

Figure 6.2 Detailed Section Through a Vessel

Other variations in testing that anticipate use conditions are possible. Polymers in actual use, loaded or stressed, are known to exhibit peculiar behavior patterns in accordance with the chemical and environmental challenge, though no difference is seen in the results of simple static (unloaded) immersion tests.

Okuda (Ref. 47) proposes that evaluation of chemical resistance be made by chemical stress relaxation. This technique is summarized in Section 5.3.26. He states that the stress relaxation test is time-saving and a superior method for evaluating chemical resistance of plastics. The stress-relaxation test is conducted directly on the sample immersed in the liquid.

Figure 6.3 below from Okuda (Ref. 47), classifies patterns of stress relaxation from which critical stress data can be derived. The critical stress data become the index of chemical degradation.

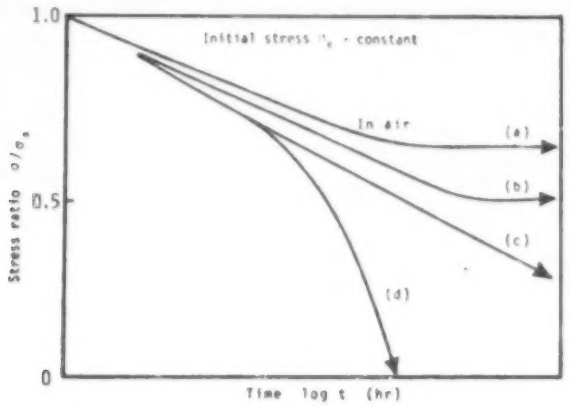


Figure 6.3 Stress Relaxation Classification

Source: Ref. 47

In Figure 6.3, the standard curve (a) in air is the basis of comparison. Usually, a relaxation limit exists, and the curve becomes concave. This relaxation limit is related to the degree of movement of molecules after a long period. Curve (b) is downward from the curve in air, and the critical initial stress decreases because of a slight swelling. However, even in (b), the swollen state, a limit similar to the relaxation limit in (a) is observed. According to Okuda (Ref. 47), plastics in this state may be used safely in the chemical agent if the allowable stress chosen is at relatively low level. Curve (c) describes the case where the molecular structure of the polymer is weakened significantly by chemical degradation, and curve (d) shows how the stress is reduced remarkably by environmental

stress cracking. If patterns (c) and (d) are observed, then the plastic or elastomer is not compatible and should not be used for the corresponding environment. We know of no commercially available apparatus for carrying out Okuda's procedure, but a test device could be devised around currently available stress/strain instrumentation.

The availability of new instrumentation expands the range of polymer testing. New test techniques may provide better compatibility characterization. For instance, dynamic mechanical analysis (DMA) is a relatively new technique for measuring fundamental properties of polymeric materials, e.g., elasticity and damping. Apparatus introduced by DuPont permits properties of material to be measured easily and automatically on a micro-processor-controlled unit (the type of DMA test is determined by the choice of software). The DMA technique separates the visco-elasticity of a material into two components of modulus--a part related to elastic modulus and a part related to the damping or viscous component. These visco-elastic measurements are very sensitive indicators of internal structure, and as such can be used to develop structure/property relationships of materials. Although DMA measurements can be made isothermally, the ability to measure visco-elastic or rheological properties as they vary with temperature makes the DMA technique especially useful for predicting end-use product performance and determining effects of aging, chemical resistance, solvation, and the like.

Figures 6.4 and 6.5 show DMA results for unreinforced 80-mil thick, commercial HDPE liner before and after exposure to Laktane solvent (a mixture of aliphatic C_7 and C_8 hydrocarbons, along with cycloparaffins and aromatics; available from Exxon). Exposure was made by suspending a sample of the HDPE in a jar of Laktane at 20°C for about 24 hours (overnight soak). The sample was then removed and evaluated by DMA for comparison. In the DMA apparatus, three properties (tensile storage modulus, tensile loss modulus, and log [tan delta]) are plotted simultaneously over the temperature range. In comparing the exposed sample to the unexposed HDPE, note the change in moduli even after only 24 hours of liquid contact. The percentage change observed at 20°C amounts to a loss of 43% for both tensile storage modulus and tensile loss modulus. This change means that visco-elastic behavior of the liner has been altered by absorption of the solvent. The significance of the change remains to be determined. This exploratory experiment suggests DMA can be applied to compatibility testing.

Resolution of the dilemma in compatibility testing is not imminent. Industry has had experience and is comfortable with relatively simple immersion or one-side exposure tests. In the United States, the NSF and EPA are the only organizations that have seriously considered--and written--a test methodology for FML. The Germans also appear to be active in FML testing. Current tests probably do not meet all criteria for determining compatibility. No test fully

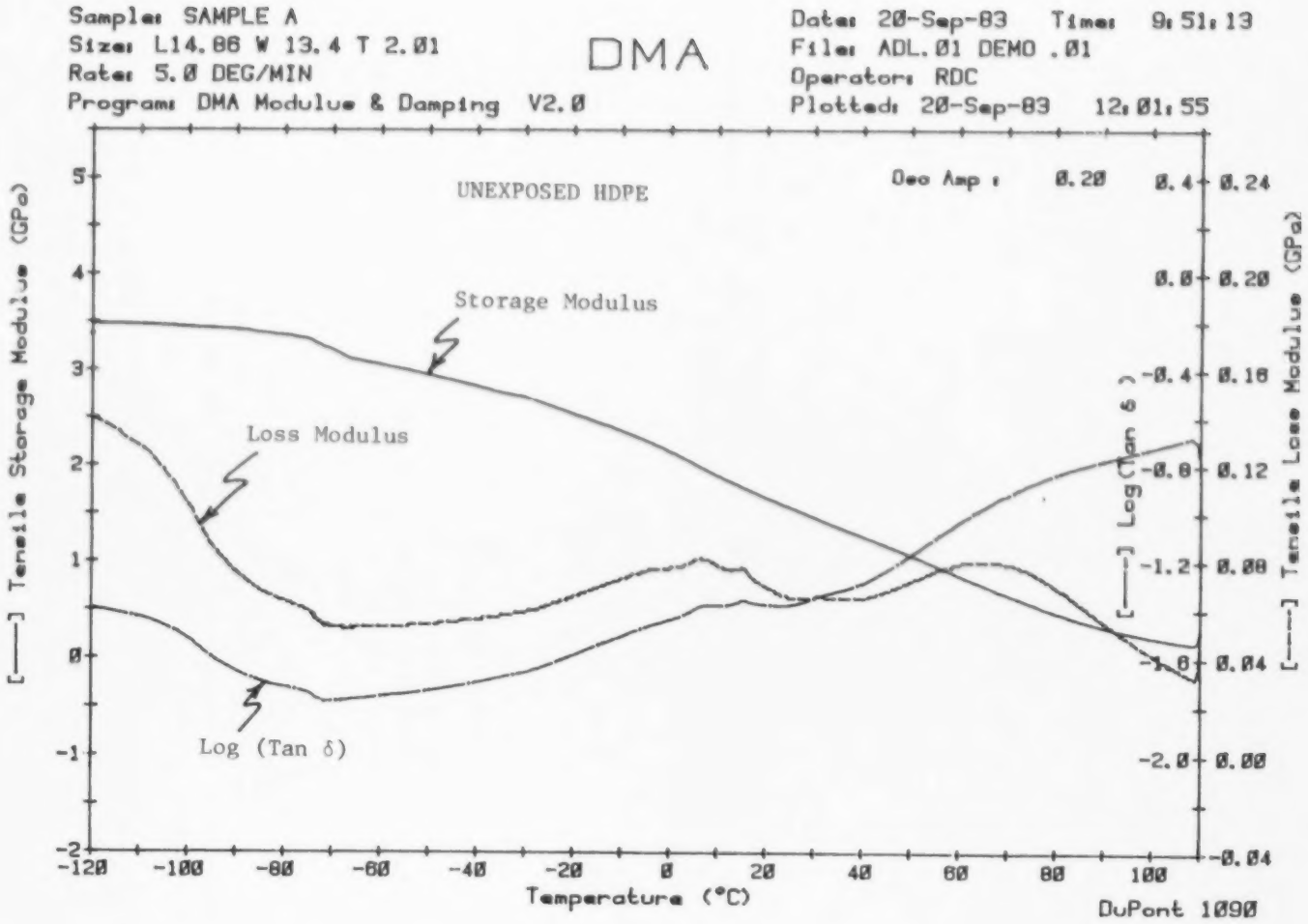


Figure 6.4 DMA - Unexposed HDPE

Source: Arthur D. Little, Inc.

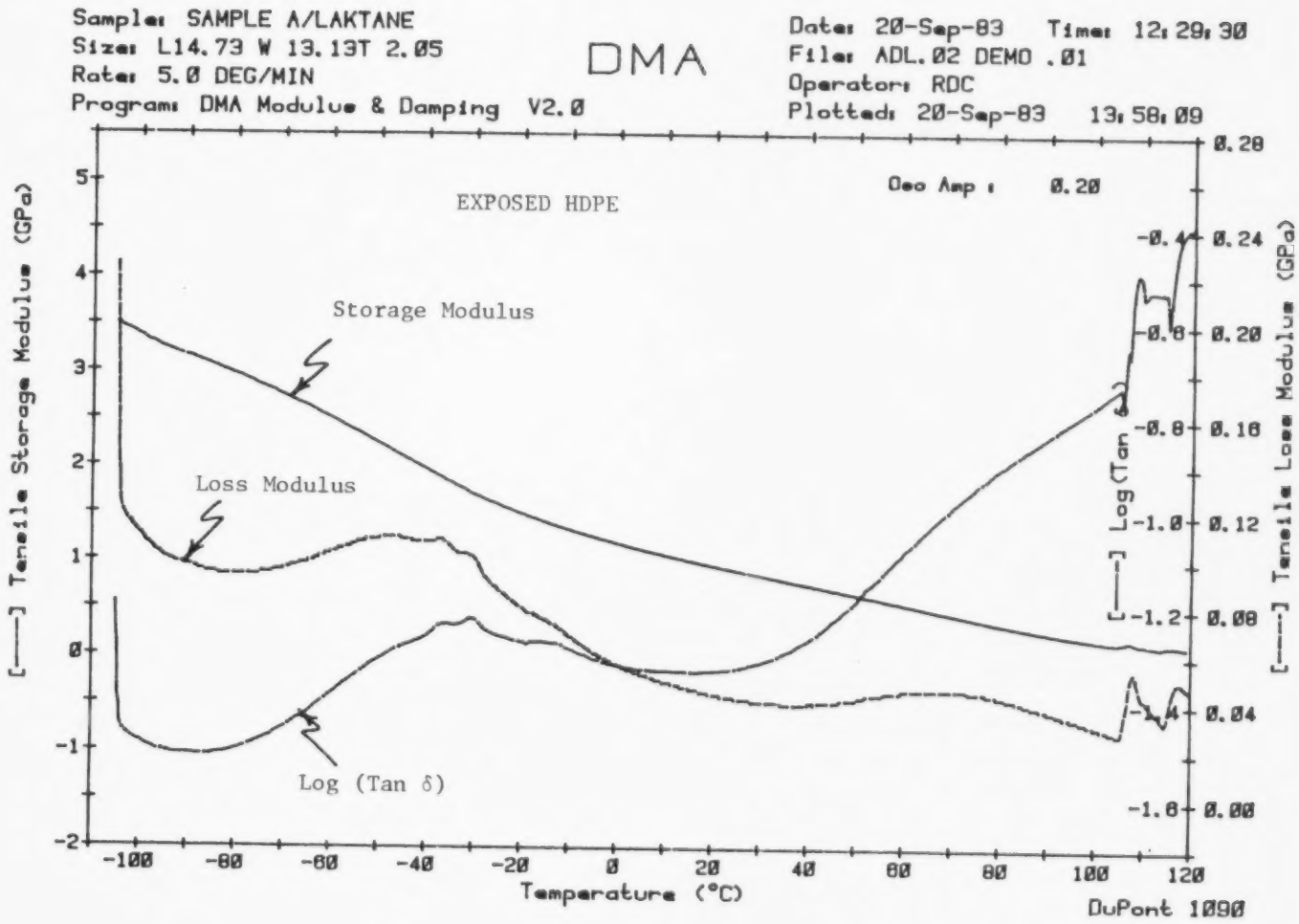


Figure 6.5 DMA - Exposed HDPE

Source: Arthur D. Little, Inc.

predicts behavior or adequately establishes life-time expectation. Laboratory screening tests do not necessarily correlate with field testing (required). The comments of FML technologists (see Section 5.6) describe as well as any the present and future test needs. Testing should be conducted at three levels to provide direction for those concerned:

1) Short term (30 days)
2) Intermediate (4 months)
3) Long term (>4 months).

Industry tests and NSF 54 deal with the short term; Method 9090 and some international tests deal with the intermediate and longer-term. None has been proven for life-time prediction or evaluation. None of the tests deals with all parameters necessary to characterize compatibility (e.g., stress-cracking, permeability and dynamic behavior).

Aside from refining present test methodology to meet immediate demands, and for building a technological and historical baseline, research is required in several areas if chemical compatibility is to be fully understood and measured. As examples, areas for investigation might include liquid transport phenomena, application of dynamic testing (e.g., visco-elastic behavior), development of long-term testing and data, and exploration of new equipment and methods for applicability. The result of current and future investigations into FML performance in a chemical environment will be a better understanding and a test(s) satisfactory to all.

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APPENDIX A

RESPONSE TO AN INQUIRY
FROM
ARTHUR D. LITTLE, INC.
BY
THE FEDERAL INSTITUTE FOR MATERIALS TESTING (BAM), BERLIN

(translated from the German)

Communication from Dr.-Ing. H. August, dated 2 November 1983, Federal Institute for Materials Testing - Bundesanstalt für Materialprufung (BAM); Elastomers, Plastics and Coating Materials Section; Unter den Eichen 87, D-1000 Berlin 45.

".....The product chemical compatibility lists published by raw material manufacturers and manufacturers of waste disposal site liners and barriers take into account only chemical effects, to the exclusion of any physical stress imposed simultaneously. Information pertains only to the effects of individual substances or chemicals on membranes, and in no way to mixtures of substances which may be present in leachate, nor the possible synergistic effects of such mixtures on polymeric membrane. These compatibility charts are therefore suitable only for initial screening and selection purposes.

"Practically, it is extremely difficult, usually impossible, to furnish precise proof of long-term suitability for a particular polymeric barrier material. In our view this is primarily attributable to the following reasons:

"The service life desired for polymeric liners (minimally 50 years and preferably 100-150 years) is beyond the scope of current test methods. All known stress-strain test procedures for testing of plastics (e.g., DIN 53 444) permit reliable extrapolation of serviceability one to 10 years or more, with maximum forecasts of up to 20 years if the duration of testing is to still remain within economically justifiable limits. Increasing test temperatures for the purpose of accelerating the test extends the extrapolation period somewhat, but this extrapolation must be applied with great care since it presupposes validity of Arrheniun behavior at the elevated temperature.

"Another difficulty is that the requirements with respect to physical and chemical stress over the life-span of the polymeric barrier is generally indeterminate. Because of the great diversity of organic and inorganic compounds that may be involved, simulation in laboratory experiments is a complex matter, is inadequate, and is poorly representative. It is known that no representative leachate from municipal waste disposal sites exists for testing membranes. The

composition of leachate is dependent on the place and location of its sampling within the waste site, varies from one disposal site to another, and is subject after sampling to aerobic and anaerobic transformations.

"Even though it follows from the reasons presented that there is no precise test method that provides, at reasonable cost and within a realistic time frame, long-term forecasts concerning the suitability of polymeric barriers, the possibility, nevertheless, exists of employing such barriers at the waste disposal site. In our view, selection should be limited to materials that have shown themselves to be outstanding over a period of ten years or more in the field of chemical storage and transport. Among the latter are high-density polyethylenes (HDPE), for which proven bonding techniques exist in tunnel and canal construction (e.g., extrusion fusing). We have no knowledge of any properties of household waste that would cause HDPE to lose its retaining and sealing capabilities for fifty years and more, so long as it is properly installed, its period of exposure to sunlight is kept as short as possible, and the PE sheeting is adequately UV-stabilized with carbon black. The environment of the PE membrane at the waste site during its period of use is largely anaerobic in our view, and this prevents significant oxidative degradation for a considerable length of time.

"When HDPE films are used in industrial waste sites, we consider necessary substantially more testing. Unless many years of experience are available as a basis for reference, substances and mixtures to be contained at the site, in concentrated form if possible, should be employed in the test. Investigations concerning possible oxidative degradation of the HDPE liner should be carried out especially when strongly oxidizing acids are present....."